

PATENT ABSTRACTS OF JAPAN

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(54) CRYSTALLIZED GLASS FOR MAGNETIC DISK SUBSTRATE, MAGNETIC DISK SUBSTRATE AND MAGNETIC DISK

(57)Abstract:

PROBLEM TO BE SOLVED: To significantly decrease the center line average surface roughness Ra of a magnetic disk substrate after the substrate is precisely polished, by increasing the Young's modulus of a crystallized glass for a magnetic disk substrate.

SOLUTION: The main crystal phase of the crystallized glass is an enstatite phase while the sub crystal phase is a magnesium aluminum titanate phase. When the peak intensity I(MS) of the enstatite phase measured by an X-ray diffraction method is 100, the peak intensity I(MAT) of the magnesium aluminum titanate phase is between ≥ 35 (preferably ≥ 40) and ≤ 70 (especially preferably ≤ 65). The Young's modulus of the glass is 120 to 155 GPa.

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 CLAIMS

[Claim(s)]

[Claim 1] Glass ceramics for magnetic-disk substrates characterized by being crystallization glass for magnetic-disk substrates, the main crystal phase of these glass ceramics for magnetic-disk substrates being an enstatite phase, a subcrystal phase being a magnesium aluminum titanate phase, peak intensity I (MAT) of the magnesium aluminum titanate phase when setting to 100 peak intensity [of the enstatite phase for which it asked with the X-ray diffraction method] I (MS) being 35 or more and 70 or less, and Young's modulus being 120-155GPa.

[Claim 2] Glass ceramics for magnetic-disk substrates according to claim 1 characterized by peak intensity I (MAT) of the magnesium aluminum titanate phase when setting to 100 peak intensity [of the enstatite phase for which it asked with the X-ray diffraction method] I (MS) being 40 or more and 65 or less.

[Claim 3] Glass ceramics for magnetic-disk substrates according to claim 1 or 2 characterized by the particle size of the crystal grain child of said glass ceramics being 0.01 micrometers - 0.1 micrometer.

[Claim 4] Glass ceramics for magnetic-disk substrates given in any one claim of claim 1-3 characterized by center line average surface roughness Ra after precision polish processing being 1-6A.

[Claim 5] The basic presentation of said glass ceramics for magnetic-disk substrates is SiO₂ : 44 - 52 % of the weight, MgO: 16-25 % of the weight, aluminum 2O₃ : 13 - 20 % of the weight, TiO₂ : 10 - 15 % of the weight, ZnO: 1- 8 % of the weight, ZrO₂ : 0 - 5 % of the weight, Li₂ O:0-3 % of the weight, B-2 O₂ : 0 - 3 % of the weight, P₂ O₅ : 0 - 5 % of the weight, and Sb 2O₃ : Glass ceramics for magnetic-disk substrates given in any one claim of claim 1-4 characterized by being 0 - 2 % of the weight.

[Claim 6] Said basic presentation of said glass ceramics for magnetic-disk substrates 2:46 - 50 % of the weight of SiO(s), MgO: 18-22 % of the weight, aluminum 2O₃ : 16 - 19 % of the weight, TiO₂ : 11 - 13 % of the weight, ZnO: 2-5%, ZrO₂ : 0 - 4 % of the weight, Li₂ O:0-2%, B-2 O₂ : 0 - 2 % of the weight, P₂ O₅ : 0 - 3 % of the weight, Sb 2O₃ : Glass ceramics for magnetic-disk substrates according to claim 5 characterized by being 0.2 - 1.5 % of the weight.

[Claim 7] The magnetic-disk substrate characterized by becoming any one claim of claims 1-6 from the crystallization glass for magnetic-disk substrates of a publication.

[Claim 8] The magnetic disk characterized by having the substrate film currently formed on the magnetic-disk substrate according to claim 7 and said magnetic-disk substrate, and a metal magnetic layer on this substrate film.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Field of the Invention] This invention relates to the magnetic-disk substrate and magnetic disk which used the crystallization glass for magnetic-disk substrates, and this.

[0002]

[Description of the Prior Art] Recently, the crystallization glass magnetic-disk substrate is examined. In glass ceramics, since most alkali-metal ion to contain exists in a crystal phase and only a minute amount exists in a glass matrix, the problem of an alkali-metal component being eluted and corroding a magnetic film is not produced.

[0003] A request that he wants to record the information especially on large capacity, such as image information, in a still smaller magnetic disk becomes strong with progress of multimedia-izing, and much more improvement in the recording density in a magnetic disk has come to be called for. In this result, especially the read/write zone of a magnetic disk, reducing center line average surface roughness (Ra) to a field 10A or less is called for.

[0004] However, in the case of glass ceramics, the degree of hardness of a crystal phase and an amorphous phase is different. For this reason, minute irregularity will occur unescapable between a crystal phase and an amorphous phase after polishing processing. Consequently, it was difficult to hold down the center line average surface roughness of a processing side to 10A or less.

[0005] It sets to JP,9-208260,A and is specific Li₂O-aluminum₂O₃-SiO₂ of a specific presentation. Center line average surface roughness Ra has tried the substrate which is 2 - 10A with the method of profit by using the magnetic-disk substrate which consists of crystallization glass of a system, and carrying out precision polish of this.

[0006]

[Problem(s) to be Solved by the Invention] However, Ra of a magnetic-disk substrate is calculated for the thing of a level (8A or less and 6 moreA or less) recently.

[0007] Moreover, in order to aim at improvement for the R/W rate of a magnetic disk, it is called for that current raises the rotational frequency which is 7200rpm to 10000-14000rpm. However, when a magnetic disk rotates at high speed, the phenomenon called the so-called flutter ring (field blurring) arises. If rotational speed becomes large, the magnitude of a flutter ring will increase remarkably.

[0008] the theoretical formula about the flutter ring when rotating a disk like a magnetic disk at high speed — various ****s — it is not established. However, for example, since [the magnitude of a flutter ring] it is in inverse proportion to Young's modulus E of the ingredient which is proportional to the square of a rotational frequency and constitutes a disk, it needs to enlarge E. Moreover, considering the calorific value of the motor at the time of high-speed rotation, the specific gravity rho of the ingredient of a disk is said for a small ingredient to be desirable. For this reason, if the rotational frequency of a magnetic disk will accelerate from now on, in order to correspond to this, an ingredient with large E/rho is indispensable.

[0009] When the central value of E/rho of each ingredient is shown, it is Li₂O-SiO₂-aluminum₂O₃ to aluminum being 27 and chemically strengthened glass being 34. System glass ceramics are 37 and are understood that the glass ceramics of this system are advantageous. However, since it corresponds to improvement in the speed of a future rotational speed, the much more big glass ceramics for magnetic-disk substrates of Young's modulus are demanded.

[0010] The technical problem of this invention is being able to be made to make small center line average surface roughness Ra after making high Young's modulus of the crystallization glass for magnetic-disk substrates and carrying out precision polish processing of the magnetic-disk substrate notably.

[0011]

[Means for Solving the Problem] This invention is the glass ceramics for magnetic-disk substrates, and the main crystal phase of this glass is an enstatite phase, a subcrystal phase is a magnesium aluminum titanate phase (it may be called a MAT phase), peak intensity I (MAT) of the magnesium aluminum titanate phase when setting to 100 peak intensity [of the enstatite phase for which it asked with the X-ray diffraction method] I (MS) is 35 or more and 70 or less, and it is characterized by Young's modulus being 120-155GPa (especially preferably 125-150 GPa).

[0012] Moreover, this invention is characterized by being the magnetic-disk substrate which consists of the aforementioned glass.

[0013] Moreover, the magnetic disk concerning this invention is characterized by having the aforementioned magnetic-disk substrate, the substrate film currently formed on it, and a metal magnetic layer on this substrate film.

[0014] As for the rate of crystallization of glass ceramics, considering as 70% or more is desirable. In addition, it says that the peak intensity of an enstatite phase is largest in an X-ray diffraction method that an enstatite phase is the main crystal phase in a crystal phase.

[0015] this invention person is SiO₂-aluminum₂O₃-MgO-TiO₂ at the process, although various kinds of quality of the materials were examined. It is system glass ceramics and succeeded in a magnesium aluminum titanate phase producing what deposits by the specific ratio as a subcrystal phase in what the main crystal phase becomes from an enstatite phase. The glass ceramics which have such crystal system are not known conventionally.

[0016] It is necessary to make into 35 or more and 70 or less peak intensity I (MAT) of the magnesium aluminum titanate phase when specifically setting to 100 peak intensity [of the enstatite phase for which it asked with the X-ray diffraction method] I (MS). The crystal in glass ceramics makes it detailed, after precision polish processing, when the smooth side whose Ra is 1 - 6A is acquired, Young's modulus E also becomes high and the thing of 120 or more GPas is obtained by this.

[0017] By making I (MAT) or more into 35 (40 or more [Especially preferably]), particle size of a crystal can be made still smaller and Ra after precision polish processing can be made still smaller. By making I (MAT) or less into 70 (65 or less [Especially preferably]), the Young's modulus of glass ceramics becomes still larger.

[0018] With the X-ray diffraction method, identification of the crystal phase of the glass ceramics of this invention might look at SiO (Mg, aluminum)3 (MAS1) and ZnAl 2O4 (Ghana Ito) other than an enstatite phase and a MAT phase. (Mg, aluminum) When I (MS) is set to 100, as for the peak intensity of each crystal phase of SiO3 (MAS1) and ZnAl 2O4 (Ghana Ito), it is desirable that it is 70 or less.

[0019] In addition, when Mg2 aluminum4 Si5 O18 (a cordierite phase, indialite), TiO2 (rutile phase), and MgOAl2 O3 SiO2 (MAS2) exist, there is an inclination for the surface roughness after precision polish to deteriorate. Therefore, an enstatite phase, a MAT phase, SiO(Mg, aluminum) 3 (MAS1), and ZnAl 2O4 When I (MS) is set to 100, as for the peak intensity of each crystal phase of an except (Ghana Ito), it is desirable that it is 20 or less.

[0020] I (MS) is obtained from the diffraction reinforcement of the peak of an enstatite phase (-0768: chemical formula MgO-SiO2 JCPDS No. 19 whenever [angle-of-diffraction] 2 theta= 31.1 degrees). I (MAT) is obtained from the diffraction reinforcement of a 2theta=25.9 degree peak whenever [angle-of-diffraction]. In this invention, a magnesium aluminum titanate phase is the generic name of each crystal phase of MgAl2 TiO 3O11 and Mg2 aluminum6 Ti 7O25. The JCPDS card number of each of these crystal phases is shown in Table 1. Whenever [angle-of-diffraction / of each / these / crystal phase], 2theta becomes 25.9 degrees or 26.1 degrees, and cannot be distinguished on an X diffraction graph.

[0021] In addition, in the patent No. 2648673 official report, the high refractoriness glass ceramics which make enstatite the main crystal phase are indicated. However, since the dissolution conditions currently indicated here are 16 hours and an elevated temperature at 1650 degrees C, the dissolution approach of common optical glass of using platinum for the wall of a crucible cannot be used for them. Consequently, after the stria in crystallization glass not disappearing but grinding a magnetic-disk substrate, it is easy to make the surface discontinuity (convex) which rose. Although the main crystal phase of this glass is an enstatite phase, other crystal phases are beta-spodumene solid solution, Ba osumilite, celsian, anorthite, a cristobalite, and a strontium feldspar.

[0022] In JP,7-53238,A, the transparent glass ceramics which make the enstatite phase whose melting temperature is 1550-1600 degrees C the main crystal phase are indicated. Since [same with the patent No. 2648673 official report] the glass ceramics currently indicated by this patent have the high melting temperature, after crystallization glass without a stria is not obtained, consequently they grind a magnetic-disk substrate, they tend to make the surface discontinuity (convex) which rose. Moreover, although the main crystal phase is an enstatite phase, the zirconia crystal phase of a minute amount exists in others.

[0023] Moreover, in JP,64-52632,A, the glass ceramics which deposit the enstatite phase which can be dissolved at 1400-1500 degrees C are indicated. However, with this crystallization glass, existence of a rutile phase is indicated with the enstatite phase. If a rutile phase exists, since the surface roughness after precision polish will worsen, it is not clearly suitable for a magnetic-disk substrate application from the above-mentioned reason. According to the place which this invention person discovered as a result of original research, it is SiO2-aluminum2 O3-MgO-TiO2. If alkaline earths other than MgO are contained in system glass ceramics, it turns out that a rutile phase becomes easy to deposit in addition to an enstatite phase. By JP,64-52632,A, since CaO is contained as an indispensable component, it is thought that the rutile phase deposits. The crystal phase of others is the Ghana Ito phase.

[0024] In JP,4-21543,A, the glass ceramics 127x10-7 / more than k are indicated for the coefficient of thermal expansion which can be dissolved at 1400 degrees C - 1500 degrees C. Existence of an enstatite phase and a MAT phase is mentioned in this crystal phase. However, with this patent reference, an enstatite phase is not the main crystal phase but SiO2. Since a phase is the main crystal phase, a detailed crystal cannot be obtained but the surface roughness after precision polish becomes large.

[0025] R. C.de VeKey And "Glass Technology" Vol.15 of A.J.Majumdar, No.3 At the June, 1974 issue, it will be CaO-aluminum2 O3-MgO-SiO2-TiO2. By the presentation of a system, evaluation with a presentation and the material property of glass ceramics is performed. However, since CaO is included, all of these examples are TiO2. The phase deposits. A rutile phase is not desirable in order to degrade the surface roughness of a precision polish object.

[0026] Said glass ceramics of this invention can be manufactured by the following approaches. That is, a basic presentation is SiO2 : 44 - 52 % of the weight, MgO:16-25 % of the weight, aluminum 2O3 : 13 - 20 % of the weight, TiO2 : 10 - 15 % of the weight, ZnO: 1-8 % of the weight, ZrO2 : 0 - 5 % of the weight, Li2 O:0-3 % of the weight, B-2 O2 : 0 - 3 % of the weight, P2 O5 : 0 - 5 % of the weight, and Sb 2O3 : 0 - 2% of the weight of parent glass is prepared.

[0027] If this parent glass is preferably crystallized at 925 degrees C - 1075 degrees C for 2 to 6 hours, the glass ceramics which make an enstatite phase the main crystal phase will be obtained. After etching glass [finishing / crystallization] by HF, when it observed under the scan mold microscope, it discovered that the crystal of enstatite existed in the magnitude of 0.01 to 0.1 micrometers.

[0028] The diameter of crystal grain decreases notably from 0.01 micrometers to 0.08 micrometers by not making it crystallize, after carrying out nucleation at the glass-transition-temperature Tg+20-Tg+50 degree C temperature currently generally especially performed by glass ceramics, but crystallizing, after carrying out nucleation in glass-transition-temperature Tg-30-Tg**0 degree C.

[0029] If precision polish of the glass ceramics [finishing / this crystallization] is carried out, the precision polish object with which Ra has especially 8A or less of smooth sides which are 1-6A will be acquired. This is considered to be because for the layer of the glass with which between crystals is filled to be [that the diameter of crystal grain is detailed, that degree of crystallinity is large, and] thin.

[0030] Mg2 aluminum4 Si 5O18 (a cordierite phase, indialite) will come to be accepted if crystallization temperature becomes 1100 degrees C or more. Moreover, MgOAl2O3 SiO2 (MAS2) will come to be accepted if crystallization temperature becomes 900 degrees C or less. Therefore, especially crystallization temperature has desirable 925-1075 degrees C.

[0031] If TiO2 (rutile phase) makes parent glass contain calcium, Sr, and Ba, it comes to deposit. The effectiveness of especially calcium is remarkable. Therefore, especially the thing that is not made to contain alkaline earth metal other than Mg is desirable.

[0032] Below, the chemical formula of each crystal phase, a name, a JCPDS card number, and an abbreviated name are shown.

[0033]

[Table 1]

化 学 式	名称	カードNo.	略称
MgSiO ₃	エンスタタイト	19-0768	MS
MgAl ₂ Ti ₂ O ₁₁	MAT	45-0450	MAT
Mg ₂ Al ₅ Ti ₇ O ₂₅	MAT	5-0451	
(Mg:Al)SiO ₃	MAS	35-0310	MAS1
Mg ₂ Al ₄ Si ₈ O ₁₈	コージュライト インド石	12-0303 13-0293	Co In
ZnAl ₂ O ₄	ガーナイト	5-0669	Ga
TiO ₂	ルチル	21-1276	T
MgOAl ₂ O ₃ SiO ₂	MAS	14-0346	MAS2
MgAl ₂ O ₄	スピネル	21-1152	Sp

[0034] The ratio of each component in parent glass is described. SiO₂ Especially the thing of an amount that it considers as 44 % of the weight or more, and is considered as 46 % of the weight or more is desirable. By this, the particle after crystallization makes it detailed. Moreover, SiO₂ By considering as 52 or less (especially preferably 50 or less % of the weight) % of the weight, the melting temperature of parent glass can be reduced and devitrification of parent glass cannot take place easily, either.

[0035] By making the amount of MgO into 16 % of the weight or more (especially preferably 18 % of the weight or more), an enstatite phase is obtained and the melting temperature of parent glass can be reduced. By making the amount of MgO into 25 or less (especially preferably 22 or less % of the weight) % of the weight, the melting temperature of glass can be reduced and devitrification of parent glass can be prevented.

[0036] aluminum 2O3 The melting temperature of glass can be reduced by making an amount into 13 % of the weight or more (especially preferably 16 % of the weight or more). aluminum 2O3 By making an amount into 20 or less (especially preferably 19 or less % of the weight) % of the weight, an enstatite phase is obtained and the melting temperature of parent glass can be reduced.

[0037] TiO₂ It becomes easy to make the particle after a crystal detailed by making an amount into 10 % of the weight or more (especially preferably 11 % of the weight or more). TiO₂ Devitrification of parent glass can be prevented by making an amount into 15 or less (especially preferably 13 or less % of the weight) % of the weight.

[0038] By making the amount of ZnO into a 1-% of the weight top (especially preferably 2 % of the weight or more), the melting temperature of parent glass becomes high. By making the amount of ZnO into 8 or less (especially preferably 5 or less % of the weight) % of the weight, devitrification of parent glass can be prevented and Vickers hardness can be made high.

[0039] Next, the arbitration component in parent glass is described. ZrO₂ The viscosity of glass can be adjusted by adding. ZrO₂ Especially the thing of an amount that it considers as 5 or less % of the weight, and is considered as 4 or less % of the weight is desirable. It can prevent that the viscosity of parent glass increases too much by this. ZrO₂ When adding, it is desirable to make the amount into 0.5 % of the weight or more.

[0040] The solubility of parent glass improves by adding Li₂ O. By making the amount of Li₂ O into 3 or less (especially preferably 2 or less % of the weight) % of the weight, the crystal grain child after crystallization makes it detailed. When adding Li₂ O, it is desirable to make the amount into 0.1 % of the weight or more.

[0041] B-2 O3 The solubility of parent glass improves by adding. B-2 O3 By making an amount into 3 or less (especially preferably 2 or less % of the weight) % of the weight, the crystal grain child after crystallization makes it detailed. B-2 O3 When adding, it is desirable to make the amount into 0.5 % of the weight or more.

[0042] P2 O5 The solubility of parent glass improves by adding. P2 O5 By making an amount into 5 or less (especially preferably 3 or less % of the weight) % of the weight, the crystal grain child after crystallization makes it detailed. P2 O5 When adding, it is desirable to make the amount into 0.5 % of the weight or more.

[0043] Sb 2O3 It acts as a defoaming agent of glass. Sb 2O3 An amount has effectiveness sufficient at 2 or less (especially preferably 0.2 to 1.5 % of the weight) % of the weight.

[0044] In case parent glass is manufactured, each raw material containing each above-mentioned metal atom is mixed so that it may correspond to the above-mentioned weight ratio, and melting of this mixture is carried out. As this raw material, the oxide of each metal atom, a carbonate, a nitrate, a sulfate, and a hydroxide can be illustrated. Moreover, as an ambient atmosphere at the time of heat-treating parent glass and making it crystallize, an atmospheric-air ambient atmosphere, reducing atmosphere, a steam ambient atmosphere, a pressurization ambient atmosphere, etc. can be chosen.

[0045] At the process which carries out precision polish processing of the material which consists of the above-mentioned glass ceramics with an abrasive grain, it grinds by the well-known precision polish processing approaches, such as the so-called wrapping and polishing, and a magnetic-disk substrate can be produced. Moreover, on the principal plane of the magnetic-disk substrate of this invention, a surface treatment layer, a magnetic film, a protective coat, etc. can be formed, and lubricant can be further applied on a protective coat.

[0046]

[Example] (Creation of parent glass) The compound containing each metal was mixed so that it might become the weight ratio of the various metallic oxides shown in Table 2, Table 3, and Table 4. It puts into the crucible made from platinum with a volume of 200 cc, and at 1430 degrees C, 250g of this mixture was heat-treated for 6 hours, and it carried out melting. After reducing the temperature of a furnace at 1300 degrees C and holding it at 1300 degrees C for 1 hour, in the mold made from carbon, it is a sink and the melt of glass was fabricated. After annealing this at 700 degrees C for 1 hour, it cooled slowly and the disc-like Plastic solid of parent glass was acquired.

[0047] From the Plastic solid of this parent glass, each tabular sample with 0.85mm [in 0.85mm in 0.85mm in dimension / of 15mm / x15mmx thickness and dimension / of 22mm / x22mmx thickness and dimension / of 5mm / x30mmx thickness] and a

dimension [of 10mm] x45mmx thickness of 1.2mm was started. In addition, each both sides of the tabular sample of thickness 0.85 (mm) and a thickness 1.2(mm) tabular sample were finish-machined with the grinding stone of #400.

[0048] (Measurement of glass transition temperature) From the tabular sample with a dimension [of 5mm] x30mmx thickness of 0.85mm, the test sample with a die length of 20mm was started. With the coefficient-of-thermal-expansion measuring device (product made from Max Science "TD5030"), the coefficient of thermal expansion of a test sample was measured in the range with a room temperature of -900 degrees C. In addition, this thermodilatometer is equipped with the function which measurement stops automatically when glass surrenders. Temperature to which the thermal-expansion curve of glass defects was made into glass transition temperature (T_g) to temperature. This value is shown in the table 2-table 4.

[0049] (Manufacture of crystallization glass) Each tabular sample was crystallized in the condition of having inserted into the carbon plate with a thickness of 5mm in nitrogen-gas-atmosphere mind. To the nucleation temperature of 720 degrees C, the temperature up of the schedule of crystallization was carried out in 300 degrees C /in an hour, and it was held at 720 degrees C for 2 hours, carried out the temperature up in 300 degrees C /from 720 degrees C to 1000 degrees C in an hour, was held at 1015 degrees C for 4 hours, and was lowered in 200 degrees C /from 1015 degrees C to the room temperature in an hour.

[0050] (Identification of a crystal phase, count of the percentage of each crystal phase) Using copper K alpha rays, X-ray diffractometer ("Geiger FREX" by Rigaku: tube voltage of 30kV, 20mA of tube electric currents) was used, and the crystal phase of the front face of the dimension 15mmx15mm tabular test sample which crystallization finished was identified. The scan include angle was performed at 2theta=2theta=15-40 degree at that time. Each detected crystal phase is shown in the table 2-table 4.

[0051] Consequently, in 1-3, the enstatite phase (-0768: chemical formula MgO-SiO₂ :JCPDS No. 19 whenever [angle-of-diffraction] 2 theta= 31.1 degrees) all deposited as a main crystal from the experimental run number 1-1 of the example of a comparison besides 1-12 from the example 1-1 of this invention, and this invention, and the MAT phase (2theta=25.9 degree) deposited as a subcrystal. Peak intensity I (MAT) of the MAT phase when setting I (MS) to 100 is shown in each table.

[0052] Moreover, the peak intensity is shown about MAS1, the Ghana Ito phase (Ga), and also other crystal phases. MAS1 is 2theta=36.0 degree whenever [angle-of-diffraction], and the Ghana Ito phase is 2theta=36.8 degree whenever [angle-of-diffraction]. Other crystal phases were also measured according to the JCPDS card of Table 1.

[0053] (Measurement of a coefficient of thermal expansion) The dimension 5mmx30mm sample which crystallization finished was cut, and the test portion with a die length of 20mm was created. With the coefficient-of-thermal-expansion measuring device (product made from a Mac science "TD5000S"), the coefficient of thermal expansion of a test sample was measured in -75 degrees C - 110 degrees C. The coefficient of thermal expansion to 100 degrees C was calculated on the basis of 25 degrees C. This value is shown in the table 2-table 4.

[0054] (Measurement of Ra in the smooth side after precision polish processing) Using the grinding stone of #700, precision grinding processing of the dimension 15mmx15mm tabular sample which identification of a crystal phase finished was carried out until the thickness of a sample was set to 0.645mm. Subsequently, using the double-sided polish board, using the high grade zirconium dioxide abrasive grain with a particle size of 0.3 micrometers, polish processing was carried out until the thickness of a sample was set to 0.635mm. Furthermore, using the colloidal silica abrasive grain with a particle size of 50A, the 2nd step of polish processings were performed and the precision polish object with a thickness of 0.635mm was acquired.

[0055] The center line average surface roughness (Ra) of the front face of a precision polish object was measured in the tapping mode of an atomic force microscope (product made from PSI "M5") using the cantilever made from silicon (resonance frequency of 300kHz). This value is shown in the table 2-table 4.

[0056] (Microstructure observation) After etching each precision polish object for 10 minutes in a fluoric acid water solution 5%, the magnitude of a crystal was observed with the scanning electron microscope. The observed diameter of crystal grain is shown in the table 2-table 4.

[0057] (Young's modulus measurement) Precision polish of the dimension 10mmx45mm tabular sample which crystallization finished was carried out in the same procedure as a dimension 15mmx15mm tabular sample. Then, after starting the dimension 4mmx40mm test portion and sticking the strain gage on this sample, the four-point bending test was performed on condition that bottom span 30mm, upper span 10mm, and crosshead speed 0.5 mm/min, and Young's modulus was calculated from the relation between a load and a variation rate.

(Measurement of specific gravity) Specific gravity was calculated from dimension 10mmx45mm the amount of water Nakashige and dry weight of a tabular sample which crystallization finished.

[0058]

[Table 2]

本発明例		1-1	1-2	1-3	1-4	1-5	1-6
酸化物重量百分率(%)	SiO ₂	46.3	45.7	47.1	47.3	47.5	48.9
	Al ₂ O ₃	17.8	17.6	15.4	18.5	20.0	17.4
	MgO	21.9	20.2	23.1	20.1	18.4	19.7
	TiO ₂	10.5	13.0	11.9	11.6	11.6	11.5
	ZnO	3.0	3.0	1.5	1.5	1.5	1.5
	Sb ₂ O ₃	0.5	0.5	1.0	1.0	1.0	1.0
ガラス転移温度 T _g (°C)		726	728	742	743	744	739
I (MAT)		45	59	44	55	63	65
I (MAS1)		47	49	46	50	47	50
I (Ga)		—	—	—	—	—	—
その他の結晶相と ピーク強度		—	—	—	—	—	—
ヤング率 (GPa)		143	138	146	136	133	132
結晶粒径 (μm)		0.05	0.02	0.06	0.04	0.03	0.02
Ra (オングスト ローム)		4	2	5	4	3	1
熱膨張係数 α × 10 ⁻⁷ /k		70	71	69	70	68	66

[0059]

[Table 3]

本発明例		1-7	1-8	1-9	1-10	1-11	1-12
酸化物重量百分率(%)	SiO ₂	44.7	46.6	46.8	47.2	46.3	46.2
	Al ₂ O ₃	19.0	17.3	18.0	17.0	17.5	17.4
	MgO	22.3	20.9	21.0	20.8	20.9	20.9
	TiO ₂	11.5	12.2	12.0	12.3	11.9	12.0
	ZnO	1.5	2.5	1.0	1.0	1.0	1.0
	Sb ₂ O ₃	1.0	0.5	0.5	0.5	0.5	0.5
	その他			Li ₂ O 0.70	B ₂ O ₃ 1.2	P ₂ O ₅ 1.9	ZrO ₂ 2.0
ガラス転移温度 T _g (°C)		758	738	729	735	737	752
I (MAT)		44	54	70	60	68	41
I (MAS 1)		57	44	55	51	48	53
I (Ga)		—	—	—	—	—	—
その他の結晶相と ピーク強度		—	—	—	—	—	—
ヤング率 (GPa)		151	134	126	135	129	140
結晶粒径 (μm)		0.08	0.05	0.04	0.04	0.02	0.04
Ra (オンゲスト ローム)		6	4	5	3	3	5
熱膨張係数 α × 10 ⁻⁷ /k		72	69	66	70	68	66

[0060]

[Table 4]

比較例		1-1	1-2	1-3
酸化物重量百分率(%)	SiO ₂	52.4	47.1	49.7
	Al ₂ O ₃	15.2	18.0	19.0
	MgO	18.3	21.9	15.3
	TiO ₂	11.6	9.5	12.1
	ZnO	1.5	3.0	1.0
	Sb ₂ O ₃	1.0	0.5	0.5
	その他			CaO 2.4
ガラス転移温度 T _g (°C)		737	728	734
I (MAT)		85	32	15
I (MAS1)		53	50	77
I (Ga)		—	—	—
その他の結晶相と ピーク強度		—	—	TiO ₂ 90
ヤング率 (GPa)		115	141	138
結晶粒径 (μm)		0.02	0.3	0.2
Ra (オングスト ローム)		5	20	10
熱膨張係数 α × 10 ⁻⁷ /k		66	72	75

[0061] The X diffraction chart of the example 1-6 of this invention is shown, the X diffraction chart of the experimental run number 1-12 in this invention is shown in drawing 2, and the X diffraction chart of the example 1-2 of a comparison is shown in drawing 1 at drawing 3. Also in other experiments, the same X diffraction chart as these has been obtained.

[0062] Young's modulus is over 125GPa(s) in any example from the example 1-1 of this invention 1-12, and the example 1-2 of a comparison and 1-3. This is considered to be the effectiveness which is considering enstatite as the main crystal.

[0063] Moreover, in the example 1-2 of a comparison, I (MAT) was 32, the diameter of crystal grain was about 0.3 micrometers, and the surface roughness after precision polish was 20A. It is TiO₂ from this. If an amount increases and the peak intensity of a MAT phase becomes large, a crystal will make it detailed. In the example 1-1 of this invention whose peak intensity of a MAT phase is 45, the surface roughness after precision polish decreased notably to 4A.

[0064] In 1-8 from an example 1-1, I (MAT) is 44-65, the particle diameter of 0.02-0.08 micrometers is obtained, and the surface roughness after precision polish becomes 6A or less.

[0065] The ceramic organization of the sample of the example 1-6 of this invention is shown in drawing 4. A detailed particle is observed.

[0066] It sets in the example 1-9 to 1-12, and is Li₂O, B₂O₃, and P₂O₅. Or ZrO₂ It is made to contain. Also in these examples, the glass ceramics whose peak intensity of a MAT phase is 35-70 were obtained by each. As for the peak intensity of a MAT phase, 45-65 is much more desirable.

[0067] In the example 1-1 of a comparison, although I (MAT) was 85, Young's modulus decreased to 115GPa(s) notably.

[0068] In the example 1-3 of a comparison, since CaO was made to contain, I (MAT) was as small as 15, the crystal was big and rough, and the surface roughness after precision polish was 10A. The photograph of the ceramic organization of the sample of the example 1-3 of a comparison is shown in drawing 5. A big and rough particle is observable.

[0069] (Measurement of Vickers hardness) The micro Vickers hardness meter was used and pushed in about the sample after precision polish, and Vickers hardness was measured by ** 1kgf. Consequently, in the example 1-6 of this invention, it was 920, was 960 in the example 1-7 of this invention, and was 770 in the example 1-1 of a comparison.

[0070] (Measurement of specific gravity) In the example 1-4 of this invention, specific gravity was 3.1g/cc. Value E/rho which broke Young's modulus E by specific gravity rho was 44.

[0071]

[Effect of the Invention] As stated above, according to this invention, center line average surface roughness Ra after making high Young's modulus of the crystallization glass for magnetic-disk substrates and carrying out precision polish processing of the magnetic-disk substrate can be notably made small.

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TECHNICAL FIELD

[Field of the Invention] This invention relates to the magnetic-disk substrate and magnetic disk which used the crystallization glass for magnetic-disk substrates, and this.

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PRIOR ART

[Description of the Prior Art] Recently, the crystallization glass magnetic-disk substrate is examined. In glass ceramics, since most alkali-metal ion to contain exists in a crystal phase and only a minute amount exists in a glass matrix, the problem of an alkali-metal component being eluted and corroding a magnetic film is not produced.

[0003] A request that he wants to record the information especially on large capacity, such as image information, in a still smaller magnetic disk becomes strong with progress of multimedia-izing, and much more improvement in the recording density in a magnetic disk has come to be called for. In this result, especially the read/write zone of a magnetic disk, reducing center line average surface roughness (Ra) to a field 10A or less is called for.

[0004] However, in the case of glass ceramics, the degree of hardness of a crystal phase and an amorphous phase is different. For this reason, minute irregularity will occur unescapable between a crystal phase and an amorphous phase after polishing processing. Consequently, it was difficult to hold down the center line average surface roughness of a processing side to 10A or less.

[0005] It sets to JP,9-208260,A and is specific Li₂O-aluminum₂O₃-SiO₂ of a specific presentation. Center line average surface roughness Ra has tried the substrate which is 2 - 10A with the method of profit by using the magnetic-disk substrate which consists of crystallization glass of a system, and carrying out precision polish of this.

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EFFECT OF THE INVENTION

[Effect of the Invention] As stated above, according to this invention, center line average surface roughness Ra after making high Young's modulus of the crystallization glass for magnetic-disk substrates and carrying out precision polish processing of the magnetic-disk substrate can be notably made small.

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TECHNICAL PROBLEM

[Problem(s) to be Solved by the Invention] However, Ra of a magnetic-disk substrate is calculated for the thing of a level (8A or less and 6 moreA or less) recently.

[0007] Moreover, in order to aim at improvement for the R/W rate of a magnetic disk, it is called for that current raises the rotational frequency which is 7200rpm to 10000-14000rpm. However, when a magnetic disk rotates at high speed, the phenomenon called the so-called flutter ring (field blurring) arises. If rotational speed becomes large, the magnitude of a flutter ring will increase remarkably.

[0008] the theoretical formula about the flutter ring when rotating a disk like a magnetic disk at high speed — various ***** — it is not established. However, for example, since [the magnitude of a flutter ring] it is in inverse proportion to Young's modulus E of the ingredient which is proportional to the square of a rotational frequency and constitutes a disk, it needs to enlarge E. Moreover, considering the calorific value of the motor at the time of high-speed rotation, the specific gravity rho of the ingredient of a disk is said for a small ingredient to be desirable. For this reason, if the rotational frequency of a magnetic disk will accelerate from now on, in order to correspond to this, an ingredient with large E/rho is indispensable.

[0009] When the central value of E/rho of each ingredient is shown, it is Li2 O-SiO2-aluminum 2O3 to aluminum being 27 and chemically strengthened glass being 34. System glass ceramics are 37 and are understood that the glass ceramics of this system are advantageous. However, since it corresponds to improvement in the speed of a future rotational speed, the much more big glass ceramics for magnetic-disk substrates of Young's modulus are demanded.

[0010] The technical problem of this invention is being able to be made to make small center line average surface roughness Ra after making high Young's modulus of the crystallization glass for magnetic-disk substrates and carrying out precision polish processing of the magnetic-disk substrate notably.

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MEANS

[Means for Solving the Problem] This invention is the glass ceramics for magnetic-disk substrates, and the main crystal phase of this glass is an enstatite phase, a subcrystal phase is a magnesium aluminum titanate phase (it may be called a MAT phase), peak intensity I (MAT) of the magnesium aluminum titanate phase when setting to 100 peak intensity [of the enstatite phase for which it asked with the X-ray diffraction method] I (MS) is 35 or more and 70 or less, and it is characterized by Young's modulus being 120-155GPa (especially preferably 125-150 GPa).

[0012] Moreover, this invention is characterized by being the magnetic-disk substrate which consists of the aforementioned glass.

[0013] Moreover, the magnetic disk concerning this invention is characterized by having the aforementioned magnetic-disk substrate, the substrate film currently formed on it, and a metal magnetic layer on this substrate film.

[0014] As for the rate of crystallization of glass ceramics, considering as 70% or more is desirable. In addition, it says that the peak intensity of an enstatite phase is largest in an X-ray diffraction method that an enstatite phase is the main crystal phase in a crystal phase.

[0015] this invention person is SiO₂-aluminum₂ O₃-MgO-TiO₂ at the process, although various kinds of quality of the materials were examined. It is system glass ceramics and succeeded in a magnesium aluminum titanate phase producing what deposits by the specific ratio as a subcrystal phase in what the main crystal phase becomes from an enstatite phase. The glass ceramics which have such crystal system are not known conventionally.

[0016] It is necessary to make into 35 or more and 70 or less peak intensity I (MAT) of the magnesium aluminum titanate phase when specifically setting to 100 peak intensity [of the enstatite phase for which it asked with the X-ray diffraction method] I (MS). The crystal in glass ceramics makes it detailed, after precision polish processing, when the smooth side whose Ra is 1 - 6A is acquired, Young's modulus E also becomes high and the thing of 120 or more GPas is obtained by this.

[0017] By making I (MAT) or more into 35 (40 or more [Especially preferably]), particle size of a crystal can be made still smaller and Ra after precision polish processing can be made still smaller. By making I (MAT) or less into 70 (65 or less [Especially preferably]), the Young's modulus of glass ceramics becomes still larger.

[0018] With the X-ray diffraction method, identification of the crystal phase of the glass ceramics of this invention might look at SiO (Mg, aluminum)₃ (MAS1) and ZnAl₂O₄ (Ghana Ito) other than an enstatite phase and a MAT phase. (Mg, aluminum) When I (MS) is set to 100, as for the peak intensity of each crystal phase of SiO₃ (MAS1) and ZnAl₂O₄ (Ghana Ito), it is desirable that it is 70 or less.

[0019] In addition, when Mg₂ aluminum₄ Si₅ O₁₈ (a cordierite phase, indialite), TiO₂ (rutile phase), and MgOAl₂ O₃ SiO₂ (MAS2) exist, there is an inclination for the surface roughness after precision polish to deteriorate. Therefore, an enstatite phase, a MAT phase, SiO(Mg, aluminum)₃ (MAS1), and ZnAl₂O₄ When I (MS) is set to 100, as for the peak intensity of each crystal phase of an except (Ghana Ito), it is desirable that it is 20 or less.

[0020] I (MS) is obtained from the diffraction reinforcement of the peak of an enstatite phase (-0768: chemical formula MgO-SiO₂ :JCPDS No. 19 whenever [angle-of-diffraction] 2 theta= 31.1 degrees). I (MAT) is obtained from the diffraction reinforcement of a 2theta=25.9 degree peak whenever [angle-of-diffraction]. In this invention, a magnesium aluminum titanate phase is the generic name of each crystal phase of MgAl₂ TiO₃ 11 and Mg₂ aluminum₆ Ti₇ O₂₅. The JCPDS card number of each of these crystal phases is shown in Table 1. Whenever [angle-of-diffraction / of each / these / crystal phase], 2theta becomes 25.9 degrees or 26.1 degrees, and cannot be distinguished on an X diffraction graph.

[0021] In addition, in the patent No. 2648673 official report, the high refractoriness glass ceramics which make enstatite the main crystal phase are indicated. However, since the dissolution conditions currently indicated here are 16 hours and an elevated temperature at 1650 degrees C, the dissolution approach of common optical glass of using platinum for the wall of a crucible cannot be used for them. Consequently, after the stria in crystallization glass not disappearing but grinding a magnetic-disk substrate, it is easy to make the surface discontinuity (convex) which rose. Although the main crystal phase of this glass is an enstatite phase, other crystal phases are beta-spodumene solid solution, Ba osumilite, celsian, anorthite, a cristobalite, and a strontium feldspar.

[0022] In JP,7-53238,A, the transparent glass ceramics which make the enstatite phase whose melting temperature is 1550-1600 degrees C the main crystal phase are indicated. Since [same with the patent No. 2648673 official report] the glass ceramics currently indicated by this patent have the high melting temperature, after crystallization glass without a stria is not obtained, consequently they grind a magnetic-disk substrate, they tend to make the surface discontinuity (convex) which rose. Moreover, although the main crystal phase is an enstatite phase, the zirconia crystal phase of a minute amount exists in others.

[0023] Moreover, in JP,64-52632,A, the glass ceramics which deposit the enstatite phase which can be dissolved at 1400-1500 degrees C are indicated. However, with this crystallization glass, existence of a rutile phase is indicated with the enstatite phase. If a rutile phase exists, since the surface roughness after precision polish will worsen, it is not clearly suitable for a magnetic-disk substrate application from the above-mentioned reason. According to the place which this invention person discovered as a result of original research, it is SiO₂-aluminum₂ O₃-MgO-TiO₂. If alkaline earths other than MgO are contained in system glass ceramics, it turns out that a rutile phase becomes easy to deposit in addition to an enstatite phase. By JP,64-52632,A, since CaO is contained as an indispensable component, it is thought that the rutile phase deposits. The crystal phase of others is the Ghana Ito phase.

[0024] In JP,4-21543,A, the glass ceramics 12x10⁻⁷ / more than k are indicated for the coefficient of thermal expansion which can be dissolved at 1400 degrees C - 1500 degrees C. Existence of an enstatite phase and a MAT phase is mentioned in this crystal phase. However, with this patent reference, an enstatite phase is not the main crystal phase but SiO₂. Since a phase is the

main crystal phase, a detailed crystal cannot be obtained but the surface roughness after precision polish becomes large.

[0025] R. C.de VeKey And "Glass Technology" Vol.15 of A.J.Majumdar, No.3 At the June, 1974 issue, it will be CaO-aluminum₂ O₃-MgO-SiO₂-TiO₂. By the presentation of a system, evaluation with a presentation and the material property of glass ceramics is performed. However, since CaO is included, all of these examples are TiO₂. The phase deposits. A rutile phase is not desirable in order to degrade the surface roughness of a precision polish object.

[0026] Said glass ceramics of this invention can be manufactured by the following approaches. That is, a basic presentation is SiO₂: 44 - 52 % of the weight, MgO: 16-25 % of the weight, aluminum₂ O₃: 13 - 20 % of the weight, TiO₂: 10 - 15 % of the weight, ZnO: 1-8 % of the weight, ZrO₂: 0 - 5 % of the weight, Li₂ O: 0-3 % of the weight, B-2 O₂: 0 - 3 % of the weight, P₂ O₅: 0 - 5 % of the weight, and Sb₂ O₃: 0 - 2 % of the weight of parent glass is prepared.

[0027] If this parent glass is preferably crystallized at 925 degrees C - 1075 degrees C for 2 to 6 hours, the glass ceramics which make an enstatite phase the main crystal phase will be obtained. After etching glass [finishing / crystallization] by HF, when it observed under the scan mold microscope, it discovered that the crystal of enstatite existed in the magnitude of 0.01 to 0.1 micrometers.

[0028] The diameter of crystal grain decreases notably from 0.01 micrometers to 0.08 micrometers by not making it crystallize, after carrying out nucleation at the glass-transition-temperature T_g+20-T_g+50 degree C temperature currently generally especially performed by glass ceramics, but crystallizing, after carrying out nucleation in glass-transition-temperature T_g-30-T_g+0 degree C.

[0029] If precision polish of the glass ceramics [finishing / this crystallization] is carried out, the precision polish object with which Ra has especially 8A or less of smooth sides which are 1-6A will be acquired. This is considered to be because for the layer of the glass with which between crystals is filled to be [that the diameter of crystal grain is detailed, that degree of crystallinity is large, and] thin.

[0030] Mg₂ aluminum₄ Si₅ O₁₈ (a cordierite phase, indialite) will come to be accepted if crystallization temperature becomes 1100 degrees C or more. Moreover, MgOAl₂O₃ SiO₂ (MAS₂) will come to be accepted if crystallization temperature becomes 900 degrees C or less. Therefore, especially crystallization temperature has desirable 925-1075 degrees C.

[0031] If TiO₂ (rutile phase) makes parent glass contain calcium, Sr, and Ba, it comes to deposit. The effectiveness of especially calcium is remarkable. Therefore, especially the thing that is not made to contain alkaline earth metal other than Mg is desirable.

[0032] Below, the chemical formula of each crystal phase, a name, a JCPDS card number, and an abbreviated name are shown.

[0033]

[Table 1]

化 学 式	名称	カードNo.	略称
MgSiO ₃	エンスタタイト	19-0768	MS
MgAl ₂ Ti ₂ O ₁₁	MAT	5-0450	MAT
Mg ₂ Al ₂ Ti ₇ O ₂₅	MAT	5-0451	
(Mg:Al)SiO ₃	MAS	35-0310	MAS1
Mg ₂ Al ₄ Si ₅ O ₁₈	コージュライト インド石	12-0303 13-0293	Co In
ZnAl ₂ O ₄	ガーナイト	5-0669	Ga
TiO ₂	ルチル	21-1276	T
MgOAl ₂ O ₃ SiO ₂	MAS	14-0346	MAS2
MgAl ₂ O ₄	スピネル	21-1152	Sp

[0034] The ratio of each component in parent glass is described. SiO₂ Especially the thing of an amount that it considers as 44 % of the weight or more, and is considered as 46 % of the weight or more is desirable. By this, the particle after crystallization makes it detailed. Moreover, SiO₂ By considering as 52 or less (especially preferably 50 or less % of the weight) % of the weight, the melting temperature of parent glass can be reduced and devitrification of parent glass cannot take place easily, either.

[0035] By making the amount of MgO into 16 % of the weight or more (especially preferably 18 % of the weight or more), an enstatite phase is obtained and the melting temperature of parent glass can be reduced. By making the amount of MgO into 25 or less (especially preferably 22 or less % of the weight) % of the weight, the melting temperature of glass can be reduced and devitrification of parent glass can be prevented.

[0036] aluminum₂ O₃ The melting temperature of glass can be reduced by making an amount into 13 % of the weight or more (especially preferably 16 % of the weight or more). aluminum₂ O₃ By making an amount into 20 or less (especially preferably 19 or less % of the weight) % of the weight, an enstatite phase is obtained and the melting temperature of parent glass can be reduced.

[0037] TiO₂ It becomes easy to make the particle after a crystal detailed by making an amount into 10 % of the weight or more (especially preferably 11 % of the weight or more). TiO₂ Devitrification of parent glass can be prevented by making an amount into 15 or less (especially preferably 13 or less % of the weight) % of the weight.

[0038] By making the amount of ZnO into a 1-% of the weight top (especially preferably 2 % of the weight or more), the melting temperature of parent glass becomes high. By making the amount of ZnO into 8 or less (especially preferably 5 or less % of the weight) % of the weight, devitrification of parent glass can be prevented and Vickers hardness can be made high.

[0039] Next, the arbitration component in parent glass is described. ZrO₂ The viscosity of glass can be adjusted by adding. ZrO₂ Especially the thing of an amount that it considers as 5 or less % of the weight, and is considered as 4 or less % of the weight is desirable. It can prevent that the viscosity of parent glass increases too much by this. ZrO₂ When adding, it is desirable to make the amount into 0.5 % of the weight or more.

[0040] The solubility of parent glass improves by adding Li₂ O. By making the amount of Li₂ O into 3 or less (especially preferably 2

or less % of the weight) % of the weight, the crystal grain child after crystallization makes it detailed. When adding Li_2O , it is desirable to make the amount into 0.1 % of the weight or more.

[0041] B-2 O3 The solubility of parent glass improves by adding. B-2 O3 By making an amount into 3 or less (especially preferably 2 or less % of the weight) % of the weight, the crystal grain child after crystallization makes it detailed. B-2 O3 When adding, it is desirable to make the amount into 0.5 % of the weight or more.

[0042] P2 O5 The solubility of parent glass improves by adding. P2 O5 By making an amount into 5 or less (especially preferably 3 or less % of the weight) % of the weight, the crystal grain child after crystallization makes it detailed. P2 O5 When adding, it is desirable to make the amount into 0.5 % of the weight or more.

[0043] Sb 2O3 It acts as a defoaming agent of glass. Sb 2O3 An amount has effectiveness sufficient at 2 or less (especially preferably 0.2 to 1.5 % of the weight) % of the weight.

[0044] In case parent glass is manufactured, each raw material containing each above-mentioned metal atom is mixed so that it may correspond to the above-mentioned weight ratio, and melting of this mixture is carried out. As this raw material, the oxide of each metal atom, a carbonate, a nitrate, a sulfate, and a hydroxide can be illustrated. Moreover, as an ambient atmosphere at the time of heat-treating parent glass and making it crystallize, an atmospheric-air ambient atmosphere, reducing atmosphere, a steam ambient atmosphere, a pressurization ambient atmosphere, etc. can be chosen.

[0045] At the process which carries out precision polish processing of the material which consists of the above-mentioned glass ceramics with an abrasive grain, it grinds by the well-known precision polish processing approaches, such as the so-called wrapping and polishing, and a magnetic-disk substrate can be produced. Moreover, on the principal plane of the magnetic-disk substrate of this invention, a surface treatment layer, a magnetic film, a protective coat, etc. can be formed, and lubricant can be further applied on a protective coat.

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EXAMPLE

[Example] (Creation of parent glass) The compound containing each metal was mixed so that it might become the weight ratio of the various metallic oxides shown in Table 2, Table 3, and Table 4. It puts into the crucible made from platinum with a volume of 200 cc, and at 1430 degrees C, 250g of this mixture was heat-treated for 6 hours, and it carried out melting. After reducing the temperature of a furnace at 1300 degrees C and holding it at 1300 degrees C for 1 hour, in the mold made from carbon, it is a sink and the melt of glass was fabricated. After annealing this at 700 degrees C for 1 hour, it cooled slowly and the disc-like Plastic solid of parent glass was acquired.

[0047] From the Plastic solid of this parent glass, each tabular sample with 0.85mm [in 0.85mm in 0.85mm in dimension / of 15mm / x15mmx thickness and dimension / of 22mm / x22mmx thickness and dimension / of 5mm / x30mmx thickness] and a dimension [of 10mm] x45mmx thickness of 1.2mm was started. In addition, each both sides of the tabular sample of thickness 0.85 (mm) and a thickness 1.2(mm) tabular sample were finish-machined with the grinding stone of #400.

[0048] (Measurement of glass transition temperature) From the tabular sample with a dimension [of 5mm] x30mmx thickness of 0.85mm, the test sample with a die length of 20mm was started. With the coefficient-of-thermal-expansion measuring device (product made from Max Science "TD5030"), the coefficient of thermal expansion of a test sample was measured in the range with a room temperature of -900 degrees C. In addition, this thermodilatometer is equipped with the function which measurement stops automatically when glass surrenders. Temperature to which the thermal-expansion curve of glass defects was made into glass transition temperature (T_g) to temperature. This value is shown in the table 2-table 4.

[0049] (Manufacture of crystallization glass) Each tabular sample was crystallized in the condition of having inserted into the carbon plate with a thickness of 5mm in nitrogen-gas-atmosphere mind. To the nucleation temperature of 720 degrees C, the temperature up of the schedule of crystallization was carried out in 300 degrees C /in an hour, and it was held at 720 degrees C for 2 hours, carried out the temperature up in 300 degrees C /from 720 degrees C to 1000 degrees C in an hour, was held at 1015 degrees C for 4 hours, and was lowered in 200 degrees C /from 1015 degrees C to the room temperature in an hour.

[0050] (Identification of a crystal phase, count of the percentage of each crystal phase) Using copper K alpha rays, X-ray diffractometer ("Geiger FREX" by Rigaku: tube voltage of 30kV, 20mA of tube electric currents) was used, and the crystal phase of the front face of the dimension 15mmx15mm tabular test sample which crystallization finished was identified. The scan include angle was performed at 2theta=2theta=15-40 degree at that time. Each detected crystal phase is shown in the table 2-table 4.

[0051] Consequently, in 1-3, the enstatite phase (-0768: chemical formula MgO-SiO₂ :JCPDS No. 19 whenever [angle-of-diffraction] 2 theta= 31.1 degrees) all deposited as a main crystal from the experimental run number 1-1 of the example of a comparison besides 1-12 from the example 1-1 of this invention, and this invention, and the MAT phase (2theta=25.9 degree) deposited as a subcrystal. Peak intensity I (MAT) of the MAT phase when setting I (MS) to 100 is shown in each table.

[0052] Moreover, the peak intensity is shown about MAS1, the Ghana Ito phase (Ga), and also other crystal phases. MAS1 is 2theta=36.0 degree whenever [angle-of-diffraction], and the Ghana Ito phase is 2theta=36.8 degree whenever [angle-of-diffraction]. Other crystal phases were also measured according to the JCPDS card of Table 1.

[0053] (Measurement of a coefficient of thermal expansion) The dimension 5mmx30mm sample which crystallization finished was cut, and the test portion with a die length of 20mm was created. With the coefficient-of-thermal-expansion measuring device (product made from a Mac science "TD5000S"), the coefficient of thermal expansion of a test sample was measured in -75 degrees C - 110 degrees C. The coefficient of thermal expansion to 100 degrees C was calculated on the basis of 25 degrees C. This value is shown in the table 2-table 4.

[0054] (Measurement of Ra in the smooth side after precision polish processing) Using the grinding stone of #700, precision grinding processing of the dimension 15mmx15mm tabular sample which identification of a crystal phase finished was carried out until the thickness of a sample was set to 0.645mm. Subsequently, using the double-sided polish board, using the high grade zirconium dioxide abrasive grain with a particle size of 0.3 micrometers, polish processing was carried out until the thickness of a sample was set to 0.635mm. Furthermore, using the colloidal silica abrasive grain with a particle size of 50A, the 2nd step of polish processings were performed and the precision polish object with a thickness of 0.635mm was acquired.

[0055] The center line average surface roughness (Ra) of the front face of a precision polish object was measured in the tapping mode of an atomic force microscope (product made from PSI "M5") using the cantilever made from silicon (resonance frequency of 300kHz). This value is shown in the table 2-table 4.

[0056] (Microstructure observation) After etching each precision polish object for 10 minutes in a fluoric acid water solution 5%, the magnitude of a crystal was observed with the scanning electron microscope. The observed diameter of crystal grain is shown in the table 2-table 4.

[0057] (Young's modulus measurement) Precision polish of the dimension 10mmx45mm tabular sample which crystallization finished was carried out in the same procedure as a dimension 15mmx15mm tabular sample. Then, after starting the dimension 4mmx40mm test portion and sticking the strain gage on this sample, the four-point bending test was performed on condition that bottom span 30mm, upper span 10mm, and crosshead speed 0.5 mm/min, and Young's modulus was calculated from the relation between a load and a variation rate.

(Measurement of specific gravity) Specific gravity was calculated from dimension 10mmx45mm the amount of water Nakashige and dry weight of a tabular sample which crystallization finished.

[0058]

[Table 2]

本発明例		1-1	1-2	1-3	1-4	1-5	1-6
酸化物重量百分率(%)	SiO ₂	46.3	45.7	47.1	47.3	47.5	48.9
	Al ₂ O ₃	17.8	17.6	15.4	18.5	20.0	17.4
	MgO	21.9	20.2	23.1	20.1	18.4	19.7
	TiO ₂	10.5	13.0	11.9	11.6	11.6	11.5
	ZnO	3.0	3.0	1.5	1.5	1.5	1.5
	Sb ₂ O ₃	0.5	0.5	1.0	1.0	1.0	1.0
ガラス転移温度 T _g (°C)		726	728	742	743	744	739
I (MAT)		45	59	44	55	63	65
I (MAS1)		47	49	46	50	47	50
I (Ga)		—	—	—	—	—	—
その他の結晶相と ピーク強度		—	—	—	—	—	—
ヤング率 (GPa)		143	138	146	136	133	132
結晶粒径 (μm)		0.05	0.02	0.06	0.04	0.03	0.02
Ra (オングスト ローム)		4	2	5	4	3	1
熱膨張係数 α × 10 ⁻⁷ /K		70	71	69	70	68	66

[0059]

[Table 3]

本発明例		1-7	1-8	1-9	1-10	1-11	1-12
酸化物重量百分率(%)	SiO ₂	44.7	46.6	46.8	47.2	46.3	46.2
	Al ₂ O ₃	19.0	17.3	18.0	17.0	17.5	17.4
	MgO	22.3	20.9	21.0	20.8	20.9	20.9
	TiO ₂	11.5	12.2	12.0	12.3	11.9	12.0
	ZnO	1.5	2.5	1.0	1.0	1.0	1.0
	Sb ₂ O ₃	1.0	0.5	0.5	0.5	0.5	0.5
	その他			Li ₂ O 0.70	B ₂ O ₃ 1.2	P ₂ O ₅ 1.9	ZrO ₂ 2.0
ガラス転移温度 T _g (°C)	758	738	729	735	737	752	
I (MAT)	44	54	70	60	68	41	
I (MAS 1)	57	44	55	51	48	53	
I (Ga)	—	—	—	—	—	—	
その他の結晶相と ピーク強度	—	—	—	—	—	—	
ヤング率 (GPa)	151	134	126	135	129	140	
結晶粒径 (μm)	0.08	0.05	0.04	0.04	0.02	0.04	
Ra (オングスト ローム)	6	4	5	3	3	5	
熱膨張係数 α ×10 ⁻⁷ /k	72	69	66	70	68	66	

[0060]

[Table 4]

比較例		1-1	1-2	1-3
酸化物重量百分率(%)	SiO ₂	52.4	47.1	49.7
	Al ₂ O ₃	15.2	18.0	19.0
	MgO	18.3	21.9	15.3
	TiO ₂	11.6	9.5	12.1
	ZnO	1.5	3.0	1.0
	Sb ₂ O ₃	1.0	0.5	0.5
	その他			CaO 2.4
ガラス転移温度 T _g (°C)		737	728	734
I (MAT)		85	32	15
I (MAS1)		53	50	77
I (Ga)		—	—	—
その他の結晶相と ピーク強度		—	—	TiO ₂ 90
ヤング率 (GPa)		115	141	138
結晶粒径 (μm)		0.02	0.3	0.2
Ra (オングスト ローム)		5	20	10
熱膨張係数 α × 10 ⁻⁷ /k		66	72	75

[0061] The X diffraction chart of the example 1-6 of this invention is shown, the X diffraction chart of the experimental run number 1-12 in this invention is shown in drawing 2, and the X diffraction chart of the example 1-2 of a comparison is shown in drawing 1 at drawing 3. Also in other experiments, the same X diffraction chart as these has been obtained.

[0062] Young's modulus is over 125GPa(s) in any example from the example 1-1 of this invention 1-12, and the example 1-2 of a comparison and 1-3. This is considered to be the effectiveness which is considering enstatite as the main crystal.

[0063] Moreover, in the example 1-2 of a comparison, I (MAT) was 32, the diameter of crystal grain was about 0.3 micrometers, and the surface roughness after precision polish was 20A. It is TiO₂ from this. If an amount increases and the peak intensity of a MAT phase becomes large, a crystal will make it detailed. In the example 1-1 of this invention whose peak intensity of a MAT phase is 45, the surface roughness after precision polish decreased notably to 4A.

[0064] In 1-8 from an example 1-1, I (MAT) is 44-65, the particle diameter of 0.02-0.08 micrometers is obtained, and the surface roughness after precision polish becomes 6A or less.

[0065] The ceramic organization of the sample of the example 1-6 of this invention is shown in drawing 4. A detailed particle is observed.

[0066] It sets in the example 1-9 to 1-12, and is Li₂O, B₂O₃, and P₂O₅. Or ZrO₂ It is made to contain. Also in these examples, the glass ceramics whose peak intensity of a MAT phase is 35-70 were obtained by each. As for the peak intensity of a MAT phase, 45-65 is much more desirable.

[0067] In the example 1-1 of a comparison, although I (MAT) was 85, Young's modulus decreased to 115GPa(s) notably.

[0068] In the example 1-3 of a comparison, since CaO was made to contain, I (MAT) was as small as 15, the crystal was big and rough, and the surface roughness after precision polish was 10A. The photograph of the ceramic organization of the sample of the example 1-3 of a comparison is shown in drawing 5. A big and rough particle is observable.

[0069] (Measurement of Vickers hardness) The micro Vickers hardness meter was used and pushed in about the sample after precision polish, and Vickers hardness was measured by ** 1kgf. Consequently, in the example 1-6 of this invention, it was 920, was 960 in the example 1-7 of this invention, and was 770 in the example 1-1 of a comparison.

[0070] (Measurement of specific gravity) In the example 1-4 of this invention, specific gravity was 3.1g/cc. Value E/rho which broke Young's modulus E by specific gravity rho was 44.

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DESCRIPTION OF DRAWINGS

[Brief Description of the Drawings]

[Drawing 1] The X diffraction chart of the example 1-6 of this invention is shown.

[Drawing 2] The X diffraction chart of the example 1-12 of this invention is shown.

[Drawing 3] The X diffraction chart of the example 1-2 of a comparison is shown.

[Drawing 4] It is the scanning electron microscope in which the ceramic organization of the sample of the example 1-6 of this invention is shown.

[Drawing 5] It is the scanning electron microscope in which the photograph of the ceramic organization of the sample of the example 1-3 of a comparison is shown.

[Translation done.]

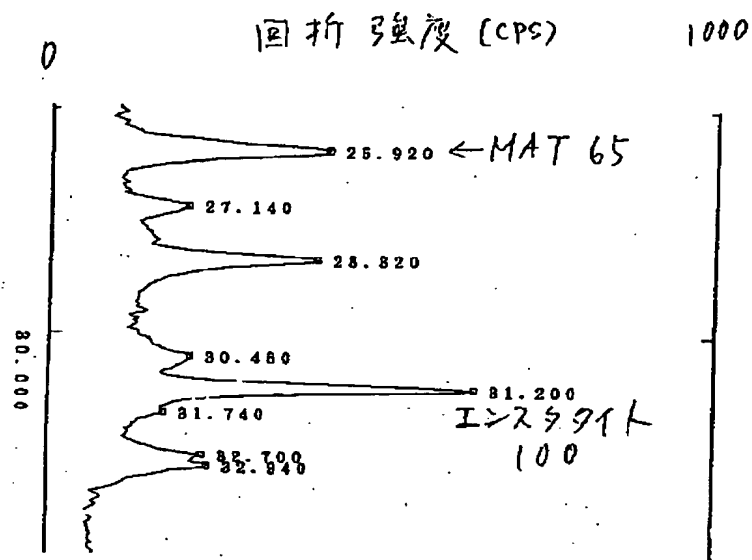
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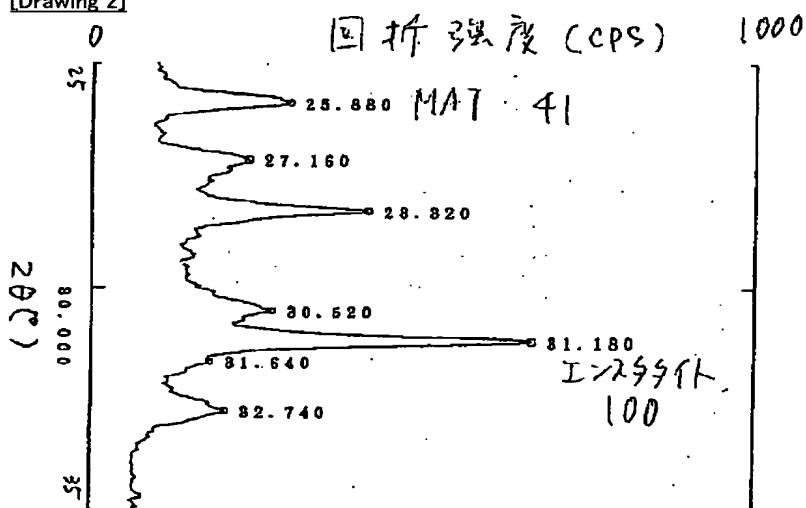
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DRAWINGS

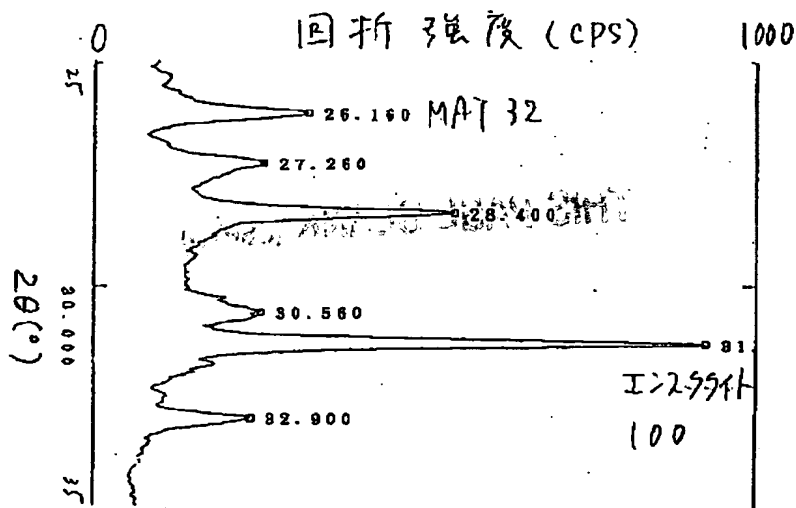
[Drawing 1]



[Drawing 2]

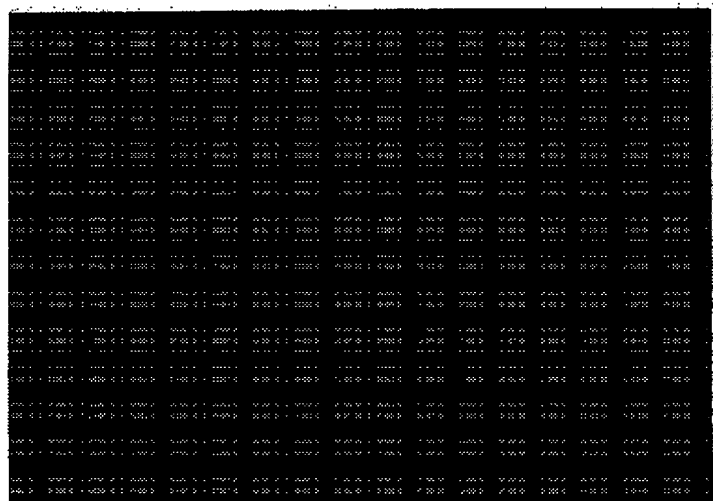


[Drawing 3]



[Drawing 4]

図面代用写真



[Drawing 5]

図面代用写真



[Translation done.]

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(54) 【発明の名称】 磁気ディスク基板用結晶化ガラス、磁気ディスク基板および磁気ディスク

(57) 【要約】

【課題】 磁気ディスク基板用結晶化ガラスのヤング率を高くし、磁気ディスク基板を精密研磨加工した後の中心線平均表面粗さ R a を顕著に小さくする。

【解決手段】 前記結晶化ガラスの主結晶相がエンスタタイト相であり、副結晶相がマグネシウムアルミニウムチタネート相であり、X線回折法で求めたエンスタタイト相のピーク強度 I (MS) を 1 0 0 としたときのマグネシウムアルミニウムチタネート相のピーク強度 I (MAT) が 3 5 以上 (特に好ましくは 4 0 以上)、7 0 以下 (特に好ましくは 6 5 以下) であり、ヤング率が 1 2 0 ~ 1 5 5 G P a である。

【特許請求の範囲】

【請求項 1】磁気ディスク基板用結晶化ガラスであって、この磁気ディスク基板用結晶化ガラスの主結晶相がエンスタタイト相であり、副結晶相がマグネシウムアルミニウムチタネート相であり、X線回折法で求めたエンスタタイト相のピーク強度 I (MS) を 100 としたときのマグネシウムアルミニウムチタネート相のピーク強度 I (MAT) が 35 以上、70 以下であり、ヤング率が 120 ~ 155 GPa であることを特徴とする、磁気ディスク基板用結晶化ガラス。

【請求項 2】X線回折法で求めたエンスタタイト相のピーク強度 I (MS) を 100 としたときのマグネシウムアルミニウムチタネート相のピーク強度 I (MAT) が 40 以上、65 以下であることを特徴とする、請求項 1 記載の磁気ディスク基板用結晶化ガラス。

【請求項 3】前記結晶化ガラスの結晶粒子の粒径が 0.01 μm ~ 0.1 μm であることを特徴とする、請求項 1 または 2 記載の磁気ディスク基板用結晶化ガラス。

【請求項 4】精密研磨加工後の中心線平均表面粗さ Ra が 1 ~ 6 オングストロームであることを特徴とする、請求項 1 ~ 3 のいずれか一つの請求項に記載の磁気ディスク基板用結晶化ガラス。

【請求項 5】前記磁気ディスク基板用結晶化ガラスの基本組成が SiO₂ : 44 ~ 52 重量%、MgO : 16 ~ 25 重量%、Al₂O₃ : 13 ~ 20 重量%、TiO₂ : 10 ~ 15 重量%、ZnO : 1 ~ 8 重量%、ZrO₂ : 0 ~ 5 重量%、Li₂O : 0 ~ 3 重量%、B₂O₃ : 0 ~ 3 重量%、P₂O₅ : 0 ~ 5 重量% および Sb₂O₃ : 0 ~ 2 重量% であることを特徴とする、請求項 1 ~ 4 のいずれか一つの請求項に記載の磁気ディスク基板用結晶化ガラス。

【請求項 6】前記磁気ディスク基板用結晶化ガラスの前記基本組成が、SiO₂ : 46 ~ 50 重量%、MgO : 18 ~ 22 重量%、Al₂O₃ : 16 ~ 19 重量%、TiO₂ : 11 ~ 13 重量%、ZnO : 2 ~ 5%、ZrO₂ : 0 ~ 4 重量%、Li₂O : 0 ~ 2%、B₂O₃ : 0 ~ 2 重量%、P₂O₅ : 0 ~ 3 重量%、Sb₂O₃ : 0.2 ~ 1.5 重量% であることを特徴とする、請求項 5 記載の磁気ディスク基板用結晶化ガラス。

【請求項 7】請求項 1 ~ 6 のいずれか一つの請求項に記載の磁気ディスク基板用結晶化ガラスからなることを特徴とする、磁気ディスク基板。

【請求項 8】請求項 7 記載の磁気ディスク基板、前記磁気ディスク基板上に形成されている下地膜、およびこの下地膜上の金属磁性層を備えていることを特徴とする、磁気ディスク。

【発明の詳細な説明】

【0001】

【発明の属する技術分野】本発明は、磁気ディスク基板用結晶化ガラス、これを使用した磁気ディスク基板およ

び磁気ディスクに関するものである。

【0002】

【従来の技術】最近、結晶化ガラス製の磁気ディスク基板が検討されている。結晶化ガラスにおいては、含有されるアルカリ金属イオンのほとんどが結晶相中に存在しており、ガラスマトリックス中には微量しか存在しないために、アルカリ金属成分が溶出して磁性膜を腐食するという問題は生じない。

【0003】マルチメディア化の進展に伴い、特に画像情報などの大容量の情報を、一層小型の磁気ディスク内に記録したいとの要望が強くなり、磁気ディスクにおける記録密度の一層の向上が求められるようになってきた。この結果、特に磁気ディスクのリードライトゾーンにおいては、中心線平均表面粗さ (Ra) を、10 オングストローム以下の領域まで低下させることが求められている。

【0004】しかし、結晶化ガラスの場合には、結晶相と非晶質相との硬度が相違している。このため、ポリッシング加工後においても、結晶相と非晶質相との間で不可避免的に微小な凹凸が発生してしまう。この結果、加工面の中心線平均表面粗さを 10 オングストローム以下に抑えることは困難であった。

【0005】特開平 9 - 208260 号公報においては、特定組成の特定の Li₂O-Al₂O₃-SiO₂ 系の結晶化ガラスからなる磁気ディスク基板を使用し、これを精密研磨することによって、中心線平均表面粗さ Ra が 2 - 10 オングストロームの基板を得ようと試みている。

【0006】

【発明が解決しようとする課題】しかし、最近では、磁気ディスク基板の Ra が 8 オングストローム以下、更には 6 オングストローム以下の水準のものが求められている。

【0007】また、磁気ディスクの読み書き速度を向上を図るために、現在は 7200 rpm である回転数を、10000 - 14000 rpm に上昇させることが求められている。しかし、磁気ディスクが高速で回転するときには、いわゆるフラッタリング (面ぶれ) と呼ばれる現象が生ずる。回転速度が大きくなると、フラッタリングの大きさは著しく増大する。

【0008】磁気ディスクのような円盤を高速で回転させたときの、フラッタリングについての理論式は、各種あり、確立していない。しかし、一例では、フラッタリングの大きさは、回転数の二乗に比例し、円盤を構成する材料のヤング率 E に反比例するとされているので、E を大きくすることが必要である。また、高速回転時のモーターの発熱量を考えると、円盤の材料の比重 ρ が小さい材料が好ましいと言われている。このため、今後、磁気ディスクの回転数が高速化すると、これに対応するためには、 E/ρ が大きい材料が必要不可欠である。

【0009】各材料の E/ρ の代表値を示すと、アルミニウムは27であり、化学強化ガラスは34であるのに対して、 $Li_2O-SiO_2-Al_2O_3$ 系結晶化ガラスは37であり、この系の結晶化ガラスが有利であることが分かる。しかし、今後の回転速度の高速化に対応するために、ヤング率の一層大きな磁気ディスク基板用結晶化ガラスが要望される。

【0010】本発明の課題は、磁気ディスク基板用結晶化ガラスのヤング率を高くし、磁気ディスク基板を精密研磨加工した後の中心線平均表面粗さ R_a を顕著に小さくできるようにすることである。

【0011】

【課題を解決するための手段】本発明は、磁気ディスク基板用結晶化ガラスであって、このガラスの主結晶相がエンスタタイト相であり、副結晶相がマグネシウムアルミニウムチタネート相(MAT相と呼ぶことがある)であり、X線回折法で求めたエンスタタイト相のピーク強度 $I(MS)$ を100としたときのマグネシウムアルミニウムチタネート相のピーク強度 $I(MAT)$ が35以上、70以下であり、ヤング率が120~155GPa(特に好ましくは125~150GPa)であることを特徴とする。

【0012】また、本発明は、前記のガラスからなる磁気ディスク基板であることを特徴とする。

【0013】また、本発明に係る磁気ディスクは、前記の磁気ディスク基板、その上に形成されている下地膜、およびこの下地膜上の金属磁性層を備えていることを特徴とする。

【0014】結晶化ガラスの結晶化率は、70%以上とすることが好ましい。なお、結晶相中で、エンスタタイト相が主結晶相であるとは、X線回折法において、エンスタタイト相のピーク強度が最も大きいことをいう。

【0015】本発明者は、各種の材質を検討していたが、その過程で、 $SiO_2-Al_2O_3-MgO-TiO_2$ 系結晶化ガラスであって、主結晶相がエンスタタイト相からなるものにおいて、副結晶相としてマグネシウムアルミニウムチタネート相が特定比率で析出しているものを作製することに成功した。このような結晶系を有する結晶化ガラスは、従来知られていない。

【0016】具体的には、X線回折法で求めたエンスタタイト相のピーク強度 $I(MS)$ を100としたときのマグネシウムアルミニウムチタネート相のピーク強度 $I(MAT)$ を、35以上、70以下とする必要がある。これによって、結晶化ガラス中の結晶が微細化し、精密研磨加工後に、 R_a が例えば1~6オングストロームの平滑面が得られる上、ヤング率 E も高くなり、120GPa以上のものが得られる。

【0017】 $I(MAT)$ を35以上(特に好ましくは40以上)とすることによって、結晶の粒径を一層小さくし、精密研磨加工後の R_a を一層小さくすることがで

きる。 $I(MAT)$ を70以下(特に好ましくは65以下)とすることによって、結晶化ガラスのヤング率が更に大きくなる。

【0018】X線回折法により、本発明の結晶化ガラスの結晶相を同定すると、エンスタタイト相およびMAT相の他には、 $(Mg, Al)SiO_3$ (MAS1)、 $ZnAl_2O_4$ (ガーナイト)が見られることがあった。 $(Mg, Al)SiO_3$ (MAS1)、 $ZnAl_2O_4$ (ガーナイト)の各結晶相のピーク強度は、 $I(MS)$ を100としたときに、70以下であることが好ましい。

【0019】なお、 Mg, Al, Si, O_2 (コージェライト相、インド石)、 TiO_2 (ルチル相)、 $MgO-Al_2O_3-SiO_2$ (MAS2)が存在すると、精密研磨後の表面粗さが劣化する傾向がある。従って、エンスタタイト相、MAT相、 $(Mg, Al)SiO_3$ (MAS1)、 $ZnAl_2O_4$ (ガーナイト)以外の各結晶相のピーク強度は、 $I(MS)$ を100としたときに、20以下であることが好ましい。

【0020】 $I(MS)$ は、エンスタタイト相(化学式 $MgO \cdot SiO_2$; JCPDS No. 19-0768: 回折角度 $2\theta = 31.1^\circ$)のピークの回折強度から得られる。 $I(MAT)$ は、回折角度 $2\theta = 25.9^\circ$ のピークの回折強度から得られる。本発明においては、マグネシウムアルミニウムチタネート相とは、 $MgAl_2TiO_6$ 、 Mg, Al, Ti, O_2 の各結晶相の総称である。これらの各結晶相のJCPDSカード番号は、表1に示す。これら各結晶相の回折角度 2θ は、25.9度または26.1度になり、X線回折グラフ上では区別できない。

【0021】なお、特許第2648673号公報では、エンスタタイトを主結晶相とする高耐火性結晶化ガラスが開示されている。しかし、ここで開示されている溶解条件は、1650℃で16時間と高温であるので、ルツポの内壁に白金を用いる、一般的な光学ガラスの溶解方法が使用できない。この結果、結晶化ガラス中の脈理が消えず、磁気ディスク基板を研磨した後に、盛り上がった表面欠陥(凸)を作りやすい。このガラスの主結晶相は、エンスタタイト相であるが、その他の結晶相は、 β -リチア輝石固溶体、Ba大隅石、重土長石、灰長石、クリストバライト、ストロンチウム長石である。

【0022】特開平7-53238号公報では、溶解温度が1550~1600℃の、エンスタタイト相を主結晶相とする透明な結晶化ガラスが開示されている。この特許に開示されている結晶化ガラスは、特許第2648673号公報と同じく、溶解温度が高いため、脈理のない結晶化ガラスが得られず、この結果、磁気ディスク基板を研磨した後に、盛り上がった表面欠陥(凸)を作りやすい。また、主結晶相はエンスタタイト相であるが、その他には微量のジルコニア結晶相が存在している。

【0023】また、特開昭64-52632号公報では、1400～1500℃で溶解可能な、エンスタタイト相を析出する結晶化ガラスが開示されている。しかし、この結晶化ガラスでは、エンスタタイト相とともにルチル相の存在が開示されている。ルチル相が存在すると、精密研磨後の表面粗さが悪くなるので、前述の理由から、磁気ディスク基板用途には明らかに適していない。本発明者が、独自の研究の結果、発見したところによると、 SiO_2 - Al_2O_3 - MgO - TiO_2 系結晶化ガラスに、 MgO 以外のアルカリ土類を含有すると、エンスタタイト相に加えてルチル相が析出しやすくなることが分かっている。特開昭64-52632号公報では、 CaO を必須成分として含有しているために、ルチル相が析出していると考えられる。その他は結晶相はガーナイト相である。

【0024】特開平4-21543号公報では、1400℃～1500℃で溶解可能な、熱膨張係数が $127 \times 10^{-7}/\text{K}$ 以上の結晶化ガラスが開示されている。この結晶相の中には、エンスタタイト相、MAT相の存在が言及されている。しかし、この特許文献では、エンスタタイト相は主結晶相ではなく、 SiO_2 相が主結晶相であるために、微細な結晶を得ることができず、精密研磨後の表面粗さが大きくなる。

【0025】R. C. de VeKey およびA. J. Majumdarの「Glass Technology」Vol. 15, No.3, 1974年6月号では、 CaO - Al_2O_3 - MgO - SiO_2 - TiO_2 系の組成で、組成と結晶化ガラスの材料特性との評価を行っている。しかし、これらの例はすべて CaO を含むために、 TiO_2 相が析出している。ルチル相は、精密研磨体の表面粗さを劣化させるため、好ましくない。

【0026】本発明の前記結晶化ガラスは、例えば以下の方法によって製造できる。即ち、基本組成が SiO_2 :44～52重量%、 MgO :16～25重量%、 Al_2O_3 :13～20重量%、 TiO_2 :10～15重量%、 ZnO :1～8重量%、 ZrO_2 :0～5重量

%、 Li_2O :0～3重量%、 B_2O_3 :0～3重量%、 P_2O_5 :0～5重量%および Sb_2O_3 :0～2重量%の親ガラスを準備する。

【0027】この親ガラスを925℃～1075℃で、好ましくは2～6時間結晶化すると、エンスタタイト相を主結晶相とする結晶化ガラスが得られる。結晶化済みのガラスをHFでエッチングした後、走査型顕微鏡で観察すると、エンスタタイトの結晶は、0.01から0.1μmの大きさで存在していることを発見した。

【0028】特に、一般的に結晶化ガラスで実行されている、ガラス転移温度 $T_g + 20 \sim T_g + 50$ ℃の温度で核形成した後に結晶化させるのではなく、ガラス転移温度 $T_g - 30 \sim T_g \pm 0$ ℃の範囲で核形成した後に結晶化することによって、結晶粒径は0.01μmから0.08μmまでに顕著に減少する。

【0029】この結晶化済みの結晶化ガラスを精密研磨すると、Raが8オングストローム以下、特に1～6オングストロームの平滑面を有する精密研磨体が得られる。これは、結晶粒径が微細であること、結晶化度が大きいこと、結晶の間を埋めるガラスの層が薄いことによるものと考えられる。

【0030】 Mg 、 Al 、 Si 、 O （コージェライト相、インド石）は、結晶化温度が1100℃以上になると、認められるようになる。また、 MgO 、 Al_2O_3 、 SiO_2 （MAS2）は、結晶化温度が900℃以下になると、認められるようになる。従って、結晶化温度は、925～1075℃が特に好ましい。

【0031】 TiO_2 （ルチル相）は、親ガラスに Ca 、 Sr 、 Ba を含有させると、析出するようになる。特に、 Ca はその効果が顕著である。従って、 Mg 以外のアルカリ土類金属を含有させないことが特に好ましい。

【0032】以下に、各結晶相の化学式、名称、JCPDSカード番号、略称を示す。

【0033】

【表1】

化学式	名称	カードNo.	略称
MgSiO_3	エンスタタイト	19-0768	MS
$\text{MgAl}_2\text{Ti}_2\text{O}_{11}$	MAT	5-0450	MAT
$\text{Mg}_2\text{Al}_5\text{Ti}_7\text{O}_{25}$	MAT	5-0451	
$(\text{Mg}:\text{Al})\text{SiO}_3$	MAS	35-0310	MAS1
$\text{Mg}_2\text{Al}_4\text{Si}_8\text{O}_{18}$	コージェライト インド石	12-0303 13-0293	Co In
ZnAl_2O_4	ガーナイト	5-0669	Ga
TiO_2	ルチル	21-1276	T
$\text{MgOAl}_2\text{O}_3\text{SiO}_2$	MAS	14-0346	MAS2
MgAl_2O_4	スピネル	21-1152	Sp

【0034】親ガラス中の各成分の比率について述べる。 SiO_2 の量は、44重量%以上とし、46重量%以上とすることが特に好ましい。これによって、結晶化後の粒子が微細化する。また、 SiO_2 を52重量%以下（特に好ましくは50重量%以下）とすることによって、親ガラスの溶解温度を低減でき、親ガラスの失透も起こりにくい。

【0035】 MgO の量を16重量%以上（特に好ましくは18重量%以上）とすることによって、エンスタタイト相が得られ、親ガラスの溶解温度を低減できる。 MgO の量を25重量%以下（特に好ましくは22重量%以下）とすることによって、ガラスの溶解温度を低減でき、親ガラスの失透を防止できる。

【0036】 Al_2O_3 の量を13重量%以上（特に好ましくは16重量%以上）とすることによって、ガラスの溶解温度を低減できる。 Al_2O_3 の量を20重量%以下（特に好ましくは19重量%以下）とすることによって、エンスタタイト相が得られ、親ガラスの溶解温度を低減できる。

【0037】 TiO_2 の量を10重量%以上（特に好ましくは11重量%以上）とすることによって、結晶後の粒子が微細化し易くなる。 TiO_2 の量を15重量%以下（特に好ましくは13重量%以下）とすることによって、親ガラスの失透を防止できる。

【0038】 ZnO の量を1重量%以上（特に好ましくは2重量%以上）とすることによって、親ガラスの溶解温度が高くなる。 ZnO の量を8重量%以下（特に好ましくは5重量%以下）とすることによって、親ガラスの失透を防止でき、ピッカース硬度を高くできる。

【0039】次に親ガラス中の任意成分について述べる。 ZrO_2 を添加することによって、ガラスの粘度を調整できる。 ZrO_2 の量は、5重量%以下とし、4重量%以下とすることが特に好ましい。これによって親ガラスの粘度が過度に増大するのを防止できる。 ZrO_2 を添加する場合には、その量を0.5重量%以上とすることが好ましい。

【0040】 Li_2O を添加することによって、親ガラスの溶解性が向上する。 Li_2O の量を3重量%以下（特に好ましくは2重量%以下）とすることによって、結晶化後の結晶粒子が微細化する。 Li_2O を添加する場合には、その量を0.1重量%以上とすることが好ましい。

【0041】 B_2O_3 を添加することによって、親ガラスの溶解性が向上する。 B_2O_3 の量を3重量%以下（特に好ましくは2重量%以下）とすることによって、結晶化後の結晶粒子が微細化する。 B_2O_3 を添加する場合には、その量を0.5重量%以上とすることが好ましい。

【0042】 P_2O_5 を添加することによって、親ガラスの溶解性が向上する。 P_2O_5 の量を5重量%以下

（特に好ましくは3重量%以下）とすることによって、結晶化後の結晶粒子が微細化する。 P_2O_5 を添加する場合には、その量を0.5重量%以上とすることが好ましい。

【0043】 Sb_2O_3 はガラスの脱泡剤として作用する。 Sb_2O_3 の量は、2重量%以下（特に好ましくは0.2-1.5重量%）で十分な効果がある。

【0044】親ガラスを製造する際には、上記の各金属原子を含有する各原料を、上記の重量比率に該当するように混合し、この混合物を熔融させる。この原料としては、各金属原子の酸化物、炭酸塩、硝酸塩、硫酸塩、水酸化物を例示することができる。また、親ガラスを熱処理して結晶化させる際の雰囲気としては、大気雰囲気、還元雰囲気、水蒸気雰囲気、加圧雰囲気等を選択することができる。

【0045】上記の結晶化ガラスからなる素材を、砥粒によって精密研磨加工する工程では、いわゆるラッピング、ポリッシング等、公知の精密研磨加工方法によって研磨し、磁気ディスク基板を作製できる。また、本発明の磁気ディスク基板の主面上には、下地処理層、磁性膜、保護膜等を形成することができ、更に保護膜上に潤滑剤を塗布することができる。

【0046】

【実施例】（親ガラスの作成）表2、表3、表4に示す、各種金属酸化物の重量比になるように、各金属を含む化合物を混合した。この混合物250gを、容積200ccの白金製ルツボに入れ、1430℃で6時間熱処理し、熔融させた。炉の温度を1300℃に低下させ、1300℃で1時間保持した後、カーボン製の型にガラスの熔融物を流しだし、成形した。これを700℃で1時間アニールした後、徐冷して、親ガラスの円盤状成形体を得た。

【0047】この親ガラスの成形体から、寸法15mm×15mm×厚さ0.85mm、寸法22mm×22mm×厚さ0.85mm、寸法5mm×30mm×厚さ0.85mm、寸法10mm×45mm×厚さ1.2mmの、各板状試料を切り出した。なお、厚さ0.85（mm）の板状試料、および厚さ1.2（mm）板状試料の各両面は、#400の砥石で仕上げ加工した。

【0048】（ガラス転移温度の測定）寸法5mm×30mm×厚さ0.85mmの板状試料から、長さ20mmの試験試料を切り出した。熱膨張率測定装置（マックスサイエンス製「TD5030」）で、試験試料の熱膨張率を、室温-900℃の範囲で測定した。なお、この熱膨張測定装置は、ガラスが屈伏した時点で、測定が自動停止する機能を備えている。温度に対して、ガラスの熱膨張曲線が偏曲する温度を、ガラス転移温度（ T_g ）とした。この値を表2-表4に示す。

【0049】（結晶化ガラスの製造）各板状試料を、窒素雰囲気中で、厚さ5mmのカーボン板に挟んだ状態で

結晶化させた。結晶化のスケジュールは、720℃の核形成温度まで、300℃/時間で昇温し、720℃で2時間保持し、720℃から1000℃まで300℃/時間で昇温し、1015℃で4時間保持し、1015℃から室温まで200℃/時間で降温した。

【0050】(結晶相の同定、各結晶相の構成比率の計算) 銅のK α 線を用い、X線回折装置(理学電機製「ガイガーフレックス」:管電圧30kV、管電流20mA)を使用して、結晶化の終わった寸法15mm \times 15mmの板状試験試料の表面の結晶相を同定した。その際、走査角度は、 $2\theta = 2\theta = 15 \sim 40^\circ$ で行った。検出された各結晶相を、表2-表4に示す。

【0051】この結果、本発明例1-1から1-12、本発明外の比較例の実験番号1-1から1-3においては、いずれも、主結晶としてエンスタタイト相(化学式MgO \cdot SiO $_2$: JCPDS No. 19-0768: 回折角度 $2\theta = 31.1^\circ$)が析出しており、副結晶としてMAT相($2\theta = 25.9^\circ$)が析出していた。I(MS)を100としたときのMAT相のピーク強度I(MAT)を、各表に示す。

【0052】また、MAS1、ガーナイト相(Ga)、更にはその他の結晶相について、そのピーク強度を示す。MAS1は回折角度 $2\theta = 36.0^\circ$ であり、ガーナイト相は回折角度 $2\theta = 36.8^\circ$ である。その他の結晶相も、表1のJCPDSカードに従って測定した。

【0053】(熱膨張係数の測定) 結晶化の終わった、寸法5mm \times 30mmの試料を切断して、長さ20mmの測定試料を作成した。熱膨張率測定装置(マックスインス製「TD5000S」)で、試験試料の熱膨張率を、-75℃ \sim 110℃の範囲で測定した。25℃を基準にして100℃までの熱膨張係数を計算した。この値を表2-表4に示す。

【0054】(精密研磨加工後の平滑面におけるRaの測定) 結晶相の同定の終わった寸法15mm \times 15mmの板状試料を、#700の研削砥石を使って、試料の厚みが0.645mmになるまで精密研削加工した。次いで、両面ポリッシュ盤を使って、粒径0.3 μ mの高純度酸化ジルコニウム砥粒を使って、試料の厚さが0.635mmになるまでポリッシュ加工した。更に、粒径50オングストロームのコロイダルシリカ砥粒を使って、2段階目のポリッシュ加工を行い、厚さ0.635mmの精密研磨体を得た。

【0055】シリコン製のカンチレバー(共振周波数300kHz)を用いた、原子間力顕微鏡(PSI製「M5」)のタッピングモードで、精密研磨体の表面の中心線平均表面粗さ(Ra)を測定した。この値を表2-表4に示す。

【0056】(微構造観察) 各精密研磨体を、5%フッ酸水溶液で10分間エッチングした後、走査型電子顕微鏡で、結晶の大きさを観察した。観察された結晶粒径を、表2-表4に示す。

【0057】(ヤング率測定) 結晶化の終わった、寸法10mm \times 45mmの板状試料を、寸法15mm \times 15mmの板状試料と同じ手順で、精密研磨した。その後、寸法4mm \times 40mmの測定試料を切り出し、この試料に歪みゲージを貼った上で、下スパン30mm、上スパン10mm、クロスヘッドスピード0.5mm/minの条件で4点曲げ試験を行い、荷重と変位の関係からヤング率を計算した。

(比重測定) 結晶化の終わった、寸法10mm \times 45mmの板状試料の水中重量および乾燥重量から、比重を計算した。

【0058】

【表2】

本発明例		1-1	1-2	1-3	1-4	1-5	1-6
酸化物重量百分率 (%)	SiO ₂	46.3	45.7	47.1	47.3	47.5	48.9
	Al ₂ O ₃	17.8	17.6	15.4	18.5	20.0	17.4
	MgO	21.9	20.2	23.1	20.1	18.4	19.7
	TiO ₂	10.5	13.0	11.9	11.6	11.6	11.5
	ZnO	3.0	3.0	1.5	1.5	1.5	1.5
	Sb ₂ O ₃	0.5	0.5	1.0	1.0	1.0	1.0
ガラス転移温度 T _g (°C)		726	728	742	743	744	739
I (MAT)		45	59	44	55	63	65
I (MAS 1)		47	49	46	50	47	50
I (Ga)		—	—	—	—	—	—
その他の結晶相とピーク強度		—	—	—	—	—	—
ヤング率 (GPa)		143	138	146	136	133	132
結晶粒径 (μm)		0.05	0.02	0.06	0.04	0.03	0.02
Ra (オングストローム)		4	2	5	4	3	1
熱膨張係数 α × 10 ⁻⁷ / K		70	71	69	70	68	66

【 0 0 5 9 】

【 表 3 】

本発明例		1-7	1-8	1-9	1-10	1-11	1-12
酸化物重量百分率(%)	SiO ₂	44.7	46.6	46.8	47.2	46.3	46.2
	Al ₂ O ₃	19.0	17.3	18.0	17.0	17.5	17.4
	MgO	22.3	20.9	21.0	20.8	20.9	20.9
	TiO ₂	11.5	12.2	12.0	12.3	11.9	12.0
	ZnO	1.5	2.5	1.0	1.0	1.0	1.0
	Sb ₂ O ₃	1.0	0.5	0.5	0.5	0.5	0.5
	その他			Li ₂ O 0.70	B ₂ O ₃ 1.2	P ₂ O ₅ 1.9	ZrO ₂ 2.0
ガラス転移温度 T _g (°C)		758	738	729	735	737	752
I (MAT)		44	54	70	60	68	41
I (MAS 1)		57	44	55	51	48	53
I (Ga)		—	—	—	—	—	—
その他の結晶相と ピーク強度		—	—	—	—	—	—
ヤング率 (GPa)		151	134	126	135	129	140
結晶粒径 (μm)		0.08	0.05	0.04	0.04	0.02	0.04
Ra (オングスト ローム)		6	4	5	3	3	5
熱膨張係数 α × 10 ⁻⁷ /K		72	69	66	70	68	66

【 0 0 6 0 】

【 表 4 】

比較例	1-1	1-2	1-3
酸化物重量百分率(%)	SiO ₂	52.4	47.1
	Al ₂ O ₃	15.2	18.0
	MgO	18.3	21.9
	TiO ₂	11.6	9.5
	ZnO	1.5	3.0
	Sb ₂ O ₃	1.0	0.5
	その他		CaO 2.4
ガラス転移温度 T _g (°C)	737	728	734
I (MAT)	85	32	15
I (MAS 1)	53	50	77
I (Ga)	—	—	—
その他の結晶相と ピーク強度	—	—	TiO ₂ 90
ヤング率 (GPa)	115	141	138
結晶粒径 (μm)	0.02	0.3	0.2
Ra (オングスト ローム)	5	20	10
熱膨張係数 α × 10 ⁻⁷ /k	66	72	75

【0061】図1に、本発明例1-6のX線回折チャートを示し、図2に本発明内の実験番号1-12のX線回折チャートを示し、図3に比較例1-2のX線回折チャートを示す。他の実験においても、これらと同様のX線回折チャートを得ている。

【0062】本発明例1-1から1-12と、比較例1-2、1-3では、ヤング率はいずれの例でも125 GPaを超えている。これは、エンスタタイトを主結晶としている効果であると思われる。

【0063】また、比較例1-2では、I (MAT) は32であり、結晶粒径が0.3 μm程度であり、精密研磨後の表面粗さは20オングストロームであった。これよりもTiO₂の量が増え、MAT相のピーク強度が大きくなると、結晶が微細化する。MAT相のピーク強度が45である本発明例1-1では、精密研磨後の表面粗

さは、4オングストロームまで顕著に減少した。

【0064】実施例1-1から1-8においては、I (MAT) は44~65であり、0.02~0.08 μmの粒子径が得られ、精密研磨後の表面粗さは、6オングストローム以下になる。

【0065】図4に、本発明例1-6の試料のセラミックス組織を示す。微細な粒子が観察される。

【0066】実施例1-9~1-12においては、Li₂O, B₂O₃, P₂O₅、またはZrO₂を含有させている。これらの例においても、いずれも、MAT相のピーク強度が35~70である結晶化ガラスが得られた。MAT相のピーク強度は、45~65が一層好ましい。

【0067】比較例1-1では、I (MAT) が85であるが、ヤング率が115 GPaに顕著に減少した。

【0068】比較例1-3では、CaOを含有させているために、I (MAT) が15と小さく、結晶が粗大であり、精密研磨後の表面粗さが10オングストロームであった。図5に、比較例1-3の試料のセラミックス組織の写真を示す。粗大な粒子が観察できる。

【0069】(ピッカース硬度の測定) 精密研磨後の試料について、マイクロピッカース硬度計を使用して、押し込み圧1 kgfでピッカース硬度を測定した。この結果、本発明例1-6では920であり、本発明例1-7では960であり、比較例1-1では770であった。

【0070】(比重の測定) 本発明例1-4では、比重は3.1 g/ccであった。ヤング率Eを比重ρで割った値E/ρは、44であった。

【0071】

【発明の効果】以上述べたように、本発明によれば、磁気ディスク基板用結晶化ガラスのヤング率を高くし、磁気ディスク基板を精密研磨加工した後の中心線平均表面粗さRaを顕著に小さくできる。

【図面の簡単な説明】

【図1】本発明例1-6のX線回折チャートを示す。

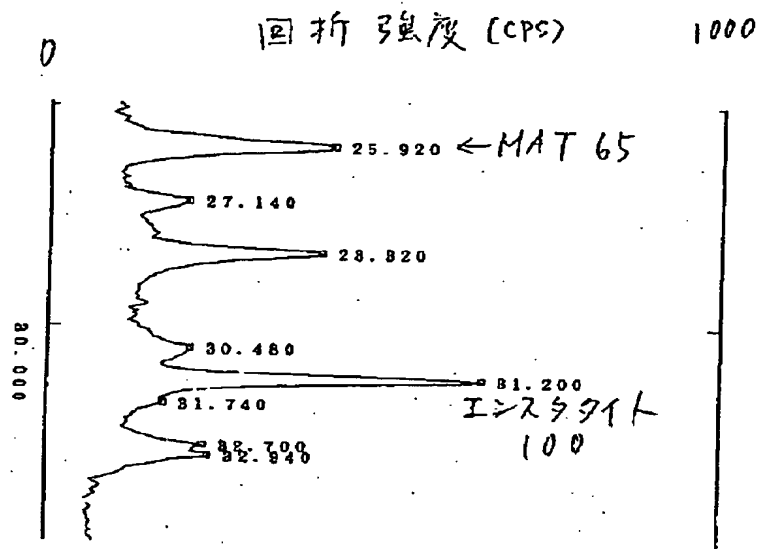
【図2】本発明例1-12のX線回折チャートを示す。

【図3】比較例1-2のX線回折チャートを示す。

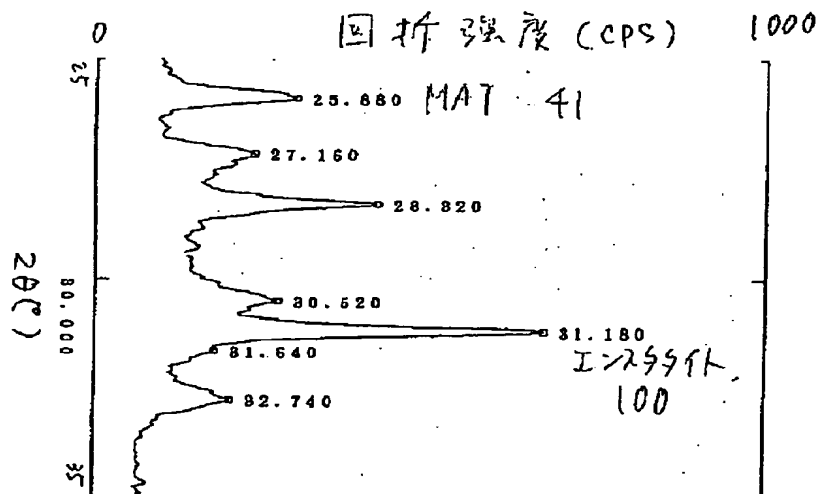
【図4】本発明例1-6の試料のセラミックス組織を示す走査型電子顕微鏡である。

【図5】比較例1-3の試料のセラミックス組織の写真を示す走査型電子顕微鏡である。

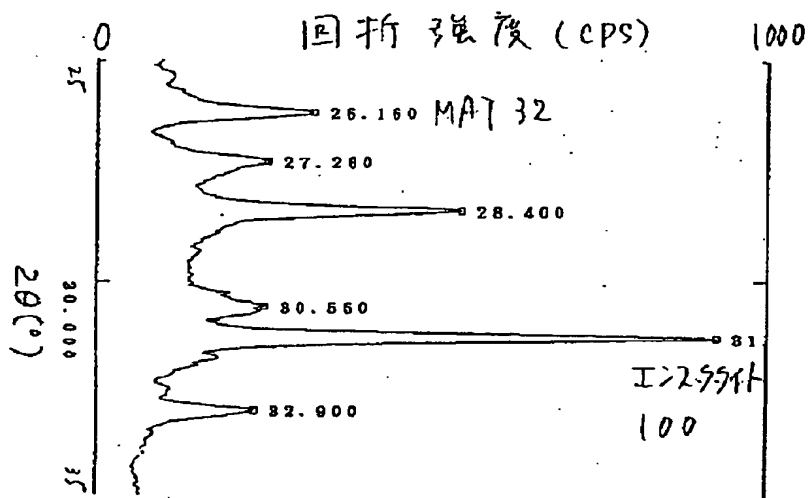
【図 1】



【図 2】

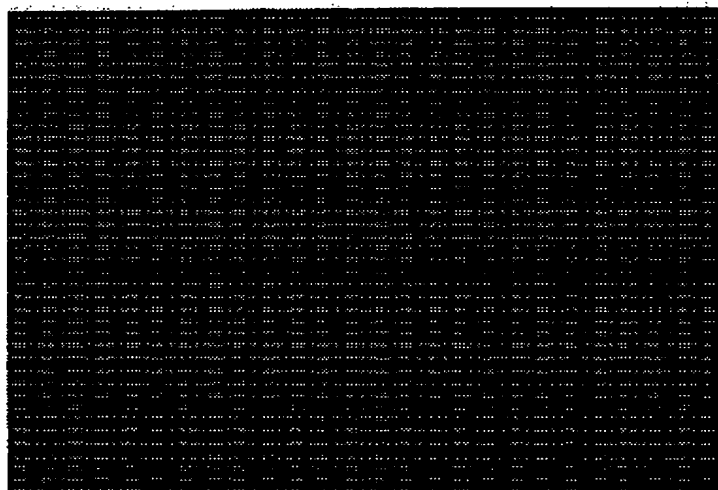


【図 3】



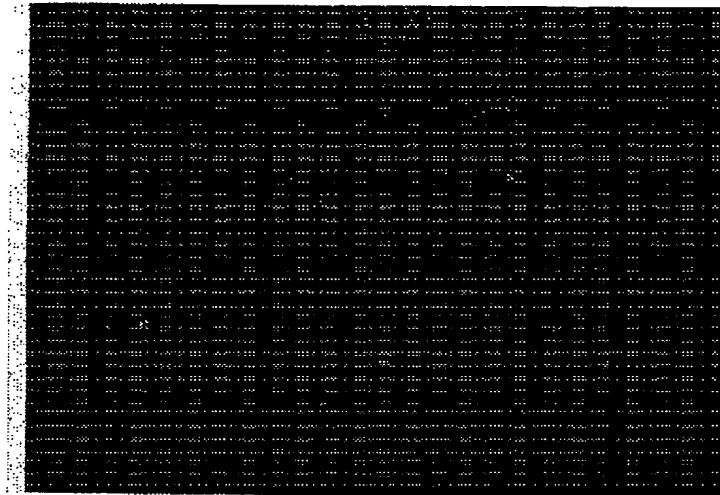
【図 4】

図面代用写真



【図 5】

図面代用写真



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